

Via Email and US Mail

20 December 2011

John Moody, US EPA Project Manager
US EPA, Region IX
Waste Management Division
75 Hawthorne Street (WST-4)
San Francisco, California 94105

Re: **RCRA FACILITY INVESTIGATION WORK PLAN**
Consent Order, Docket No RCRA (AO)-09-2008-03
Former Romic Environmental Technologies Corp. Facility
Chandler, Arizona

Dear Mr. Moody:

Pursuant to paragraphs 35, 36, and 37 of the above-referenced Administrative Order on Consent, Romic Environmental Technologies Corp. (“Romic”) is transmitting the enclosed RCRA Facility Investigation (RFI) Work Plan prepared by Clear Creek Associates, Inc. and Iris Environmental.

This RFI Work Plan reflects the scoping discussions held with USEPA and GRIC DEQ on June 21-22, 2011 and subsequent communications integrated into RFI Work Plan overview submitted on August 12, 2011.

Requested or required components of an RFI work plan were specified in the November 19, 2007 Administrative Order on Consent, and included in part a Community Relations Plan and a Communications Plan. Since USEPA has already prepared both plans during the TRIAD phase of investigation for the North Central Plume, Romic would like to continue to support USEPA ongoing efforts for the Community and utilize the existing plans RFI implementation.

Romic is prepared to implement the tasks described in the RFI Work Plan upon USEPA approval. Please do not hesitate to contact me at (510)-834-4747 x21 or calger@irisenv.com if you have any questions or comments regarding this submittal.

Sincerely,

IRIS ENVIRONMENTAL

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Principal Engineering Geologist

cc: Glenn Stark, GRIC – DEQ
Esther Manuel, LBIDC
Wayne Kiso, Clarus Management Solutions

**RCRA FACILITY INVESTIGATION WORK PLAN
FORMER ROMIC ENVIRONMENTAL TECHNOLOGIES CORP FACILITY
LONE BUTTE INDUSTRIAL PARK
GILA RIVER INDIAN COMMUNITY**



Prepared for:
ROMIC ENVIRONMENTAL TECHNOLOGIES CORP.

Prepared by:
Clear Creek Associates, PLC

December 2011

**RCRA FACILITY INVESTIGATION (RFI) WORK PLAN
FORMER ROMIC ENVIRONMENTAL TECHNOLOGIES CORP FACILITY
LONE BUTTE INDUSTRIAL PARK
GILA RIVER INDIAN COMMUNITY, ARIZONA**

Prepared For:

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**December 2011
Project No. 212001**

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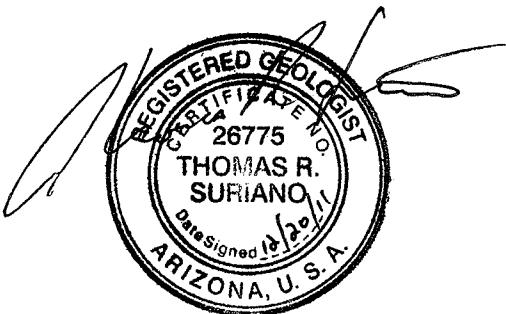


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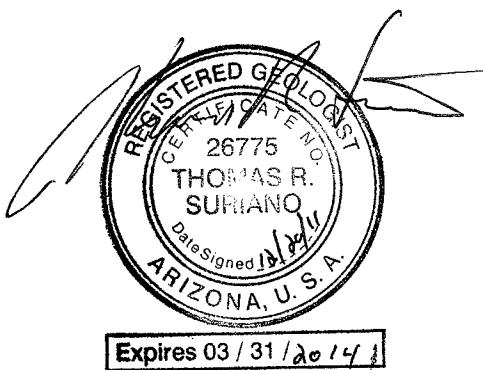
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LIST OF ACRONYMS

ADWR	Arizona Department of Water Resources
ADOT	Arizona Department of Transportation
AOC	Administrative Order on Consent
bgs	below ground surface
CA/FO	Consent Agreement / Final Order
cis-1,2-DCE	cis-1,2-dichloroethene
cis-1,3-DCP	cis-1,1-dichloropropene
Clear Creek	Clear Creek Associates, PLC
COC or COCs	chemical(s) of concern
CMS	Corrective Measures Study
CSM	Conceptual Site Model
1,1-DCE	1,1-dichloroethene
EPA	U.S. Environmental Protection Agency
GRIC	Gila River Indian Community
GRIC-DEQ	Gila River Indian Community Department of Environmental Quality
HSP	Health and Safety Plan
ICAM	Interim corrective action measures
I-10	Interstate 10
Iris	Iris Environmental
LAU	Lower Alluvial Unit
LBIDC	Lone Butte Industrial Development Corporation
MAU	Middle Alluvial Unit
MCL	Maximum Contaminant Level
MNA	Monitored natural Attenuation
PCE	tetrachloroethene
PDB	passive diffusion bag
perched zone	perched saturated zone
PID	photoionization detector
PVC	polyvinyl chloride
QAPP	Quality assurance project plan
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
Romic	Romic Environmental Technologies Corporation
SAP	Sampling and Analysis Plan
SWMU or SWMUs	solid waste management unit(s)
Southwest Solvents	Southwest Solvent Industrial Recycling
SRV	Salt River Valley

SVE	soil vapor extraction
SVOC or SVOCS	semi-volatile organic compound(s)
TAT	turnaround time
TCE	trichloroethene
TSD	treatment storage or disposal
UAU	Upper Alluvial Unit
µg/L	micrograms per liter
USCS	Unified Soils Classification System
VOA vials	volatile organic analysis vials
VOC or VOCs	volatile organic compound(s)

1.0 INTRODUCTION

This Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Work Plan has been prepared in response to the November 19, 2007 Administrative Order on Consent (AOC) between the United States Environmental Protection Agency (EPA) and Romic Environmental Technologies Corp. (Romic), Docket No RCRA (AO)-09-2008-03. The RFI Work Plan was prepared by Clear Creek Associates, PLC (Clear Creek) and Iris Environmental (Iris) pursuant to paragraphs 35, 36, and 37 of the AOC and EPA's letter dated July 6, 2011 (as amended by EPA's email of August 9, 2011). This RFI work plan was prepared to conduct work at and in the vicinity of the former Romic facility – a former waste recycling facility located in the Lone Butte Industrial Park in the Gila River Indian Community (GRIC) near Chandler, Arizona (Figure 1).

1.1 RFI WORK PLAN SCOPE AND OBJECTIVES

As stated in the AOC and EPA guidance, the focus of the RFI is to identify the nature and extent of releases of hazardous waste and/or hazardous constituents at or from the former Romic facility which may pose an unacceptable risk to human health and the environment. To date, Romic has conducted numerous investigations of the nature and extent of releases at the former facility and implemented interim corrective action measures (ICAM) to mitigate soil impacts, and has participated in TRIAD style investigations both onsite and offsite to further characterize the nature and extent of vapor and groundwater impacts. The results of the investigations and corrective measures are summarized in Section 3.0 of this Work Plan and documented in detail in the Current Conditions Report (Clear Creek Associates, 2008b) and the Conceptual Site Model (CSM) report (Clear Creek Associates, 2011a). As a continuation of this prior work, this RFI is targeted at resolving remaining investigation data gaps and collecting data considered necessary for assessing the need for additional corrective action.

Groundwater data gaps were discussed during the June 21-22, 2011 meetings with EPA and the GRIC Department of Environmental Quality (GRIC-DEQ). The core questions for the groundwater data gaps was whether the northern VOC plume extends deeper than the existing

monitoring network, extends north of well HC-09, and is adequately defined on its distal (western) end. Additional questions considered in the development of this RFI Work Plan include whether the northern VOC plume is attenuating naturally as suggested by data trends, and whether an excessive vapor risk is present at or near the former Romic facility.

As discussed further in Sections 5 and 6, Romic has developed the following tasks to complete the RFI:

- Multi-depth sampling of existing Romic and GRIC groundwater monitoring wells. (Sect. 5.2)
- Drill and sample three groundwater borings in a north-south transect west of Interstate 10 (I-10). (Sect. 6.2.1)
- Possible conversion of one or more transect borings into a monitor well. (Sect. 6.5)
- Drill and sample one supplemental groundwater boring east of I-10 between LB-4 and LB-5. (Sect. 6.2.2)

Drill and sample one geotechnical boring at the former Romic facility to establish site specific criteria for tier 2 vapor risk screening. (Sect. 6.2.3)

1.2 PURPOSE AND ORGANIZATION

The purpose of the RFI Work Plan is to establish the procedures for conducting additional investigations to address the identified groundwater data gaps. The RFI Work Plan is organized as follows

- Section 2.0 provides a brief overview of the Environmental Setting of the former Romic facility that is described in more detail in the Conceptual Site Model report (Clear Creek Associates, 2011a).
- Section 3.0 presents a brief overview of the historical investigation and IRMs

implemented on and in the vicinity of the former Romic facility that are described in detail in the Conceptual Site Model report (Clear Creek Associates, 2011a) and the Current Conditions Report (Clear Creek Associates, 2008b).

- Section 4.0 presents a brief overview of potential receptors that are described in more detail in the Conceptual Site Model report (Clear Creek Associates, 2011a).
- Section 5.0 describes the approach for conducting continued water level measurement and water quality monitoring activities.
- Section 6.0 presents the approach for performing the groundwater investigation activities and vapor risk screening to address the identified data gaps.
- Section 7.0 identifies the Preliminary Corrective Measures that will be evaluated to address the identified soil and groundwater impacts.
- Section 8.0 identifies the key personnel responsible for management of the project.
- Section 9.0 presents a schedule for implementing the work set forth in this RFI Work Plan.
- Section 10.0 identifies the References cited in this RFI Work Plan.

An updated Sampling and Analysis Plan (SAP) is provided in Appendix A of this RFI Work Plan. The updated SAP modifies the existing procedures being used for on-going groundwater monitoring activities at the former Romic facility. A revised Quality Assurance Project Plan, including data management procedures, is provided as Appendix B. An updated Health and Safety Plan covering the additional tasks being implemented to address the identified data gaps, is included as Appendix C.

2.0 ENVIRONMENTAL SETTING

This section provides a brief overview of the hydrogeologic setting of the former Romic facility including the regional geology, the local geology and the local hydrogeology. A detailed discussion of the available data and the development of the conceptual model for the site is included in the Conceptual Site Model report (Clear Creek Associates, 2011a).

2.1 REGIONAL GEOLOGY

The former Romic facility is located in the southern part of the East Salt River Valley (SRV) in the Basin and Range Physiographic Province. The Basin and Range Province is generally characterized by isolated north to northwest oriented mountain ranges separated by broad alluvial valleys. As part of the documentation for the SRV Regional Groundwater Flow Model, the Arizona Department of Water Resources (ADWR) describes three main alluvial units overlying bedrock in the Salt River Valley (Corell and Corkhill, 1994). These layers are the Upper Alluvial Unit (UAU), Middle Alluvial Unit (MAU), and Lower Alluvial Unit (LAU). The UAU is composed of mainly silt, sand, and gravel, extending from land surface to approximately 300 feet below ground surface (bgs) in the vicinity of the Romic facility. The MAU consists of mainly clay, silt, sand, and gravel with some interbedded basalt flows, extending from approximately 300 feet to over 700 feet bgs. The LAU includes clays, silts, mudstone, siltstone, sand, and gravel, and extends from below 700 feet to 1,200 feet bgs. ADWR predicts bedrock to occur below 1,200 feet bgs in parts of the SRV.

2.2 LOCAL GEOLOGY

The general lithology beneath the former Romic facility can be described by the sediments encountered during the drilling of groundwater monitor wells and soil vapor extraction (SVE) wells on the former Romic facility. The uppermost sediments, from below the surface fill to depths of approximately 60 feet bgs, are predominantly clay and silt with laterally discontinuous layers of horizontal calcareous cementation and caliche. The upper sediments are underlain by silty sand and sand from approximately 60 feet to 75 feet bgs and by silty

gravel and gravel from 75 feet to 100 feet bgs (Clear Creek Associates 2008a and 2009e, LFR 2009a). The observed lithology at SVE-02, which was advanced to 222 feet bgs, indicated alternating layers of clayey gravel and clayey sand from 100 feet to 222 feet bgs (LFR, 2009a).

The Lone Butte supply wells (A-1 and A-2) were drilled to depths in excess of 900 feet bgs. Driller's logs for A-1 and A-2 indicate that from land surface to approximately 290 feet bgs the sediments are predominantly gravels with some clays and predominantly clays below 290 feet bgs. From these driller's logs, it is estimated that the transition from the UAU to the MAU occurs at approximately 290 feet bgs. The driller's logs also indicate that the predominantly fine grained MAU extends for approximately 400 feet with the transition to the coarser grained LAU occurring at approximately 700 feet bgs.

2.3 LOCAL HYDROGEOLOGY

Groundwater at the site exists in two water-bearing zones: a perched zone, encountered within the range of approximately 40 to 60 feet bgs and the regional aquifer, generally encountered in the sand and gravel unit beginning at approximately 73 feet bgs. Chemical and hydrogeologic patterns indicate the two water-bearing zones are not directly linked.

2.3.1 Perched Zone

Inconsistent, and sporadic saturated conditions were encountered above the regional water table in a thin zone within the range of approximately 40 to 60 feet bgs beneath the former Romic facility during the monitor well and SVE well installations. A perched zone has also reportedly been identified at shallower depths at the Kinder Morgan site located northwest of the former Romic facility (verbal communication, LFR 2009). Although cascading water has been reported in GRIC DEQ monitor well LB-6 (located approximately 800 feet northeast of the former Romic facility), indicating that a shallow saturated zone exists at that location, the lateral extent of this shallow saturated zone and its relationship to the perched zone at Romic is unknown. Neither audible indications of cascading water nor spurious readings in the

electronic water level sounder have been reported at LB-4, located to the southwest of the former Romic facility, suggesting that the perched zone may not extend to the south of the former Romic facility. No lithologically distinct layer (such as a fine-grained silt or clay lens) was observed in the soil borings logged during the on-site facility investigations that would explain the presence of the perched zone. The driller's log for Lone Butte supply well A-1 dated September 8, 1968, indicated water was encountered at 31 feet bgs. However, insufficient historical water level monitoring data are available to determine if this is representative of a historically higher water table in the regional aquifer in 1968. It is currently unknown if the shallow saturated zone is related to delayed drainage from past nearby agricultural irrigation north of supply well A-1, or if the shallow saturated zone is related directly to infiltration from the adjacent canal and/or the Arizona Department of Transportation (ADOT) retention basin near LB-6. The full lateral extent of this shallow saturated zone is unknown, however, chemical impacts to the perched zone beneath the former Romic facility are dissimilar to the regional aquifer chemistry, and suggest that the perched zone is not markedly interconnected with the regional aquifer.

2.3.2 Regional Aquifer

Regional groundwater occurs in unconfined conditions at a depth of approximately 70 feet bgs. Groundwater flow directions in the regional aquifer, based on the monitoring data collected from the Romic monitor wells (RE101 through RE109) between August 2007 and August 2010, are generally west-southwest (Clear Creek Associates, 2010b). The historical data demonstrate that water levels in the regional aquifer have risen approximately 6 feet since 2007 in the vicinity of the former Romic facility. With the exception of monitoring well LB-6, the observed water level rise is generally consistent in all monitoring wells and has not had a significant influence on interpreted groundwater flow directions.

3.0 SITE HISTORY AND CHARACTERIZATION

The facility began operations in 1975 as Southwest Solvents and later as Southwest Solvent Industrial Recycling. For purposes of this work plan, these operations are collectively referred to as Southwest Solvents. Primary operations at Southwest Solvents consisted of hazardous waste recycling. While operating as Southwest Solvents, the majority of the site was unpaved, with the exception of the loading dock, the main building and four to five isolated material handling areas (Booz, Allen, Hamilton, November 2004). Following several EPA site inspections, Southwest Solvents was notified of numerous violations including unsatisfactorily operating a facility generating hazardous waste, unsatisfactorily operating a treatment storage or disposal (TSD) facility, and not furnishing information in conjunction with an interim status facility application. Based on these violations, EPA issued a Consent Agreement / Final Order (CA/FO) (RCRA-09-88-0002) to Southwest Solvents to investigate and remediate releases and to dispose of excess waste at their facility. Shortly thereafter in 1988, Romic purchased the facility out of bankruptcy from Southwest Solvents.

Romic operated under interim status since 1988 when they purchased the facility from Southwest Solvents until it shut down in 2007. In January 1999, Romic was acquired by U.S. Liquids, and subsequently, acquired by ERP Environmental Services, Inc. in August 2003. The facility operated under interim status as Romic Environmental Technologies Corporation. In November 2007, Romic ceased operations at the facility after a RCRA Part B permit was not granted for the facility.

3.1 HISTORICAL INVESTIGATIONS

Multiple site investigation programs have been conducted at and around the former Romic facility by various investigators. Additional information regarding the previous investigations and upgrades at the facility is presented in Clear Creek's August 2008 *Current Conditions Report* and Clear Creek's May 2011 *Conceptual Site Model Report*. A brief summary of these

investigations is presented below.

- **AUGUST 1987 KLEINFELDER PROPERTY TRANSFER INVESTIGATION**

Kleinfelder conducted a soil sampling investigation of existing site conditions prior to purchase of the facility by Romic. The investigation consisted of drilling eight soil borings to a depth of 20 feet bgs and collected soil samples for volatile organic compounds (VOCs) analysis by EPA Method 8010/8020. Soil analytical results detected tetrachloroethene (PCE), trichloroethene (TCE), 1,1,1-trichloroethane (1,1,1-TCA), and 1,2-dichlorobenzene (1,2-DCB) concentrations above the laboratory reporting limits in four of the borings.

- **JULY – NOVEMBER 1988 ROMIC SOIL INVESTIGATION**

Romic conducted multiple surface to near surface soil sampling investigations at the facility prior to construction and upgrade activities. The investigations consisted of collecting soil samples at depths ranging from 1 foot to 3 feet bgs. Soil samples were analyzed for VOCs by EPA Method 8010/8020.

In July 1988, 36 samples were collected from a depth of 1 foot bgs in the northern portion of the facility prior to construction of the drum storage facility. VOC concentrations were not detected above the laboratory reporting limits in 35 of the 36 soil samples collected. PCE and TCE were detected in one sample at a concentration of 0.01 milligrams per kilogram (mg/kg) (HLA, December 1989).

In August and October 1988, soil samples were collected at depths ranging from 1 foot to 3 feet bgs from 11 sample points in the eastern portion of the facility prior to construction of the secondary drum storage facility. PCE concentrations were detected above the laboratory reporting limits at five sample points to depths of 3 feet bgs. Additionally 1,1,1-TCA was detected from samples collected from two sampling points and carbon tetrachloride was detected in a sample collected from one sampling point.

In November 1988, soil samples were collected from 32 sample points south of the proposed drum storage facility at depths ranging from 1 foot to 3 feet bgs. PCE concentrations were detected above the laboratory reporting limit in 5 samples to depths of 3 feet bgs. Additionally, TCE and DCB concentrations were detected above the laboratory reporting limits at two sample locations and xylene concentrations were detected above the laboratory reporting limit at one sample location.

- **FEBRUARY 1989 HLA SHALLOW SOIL INVESTIGATION**

Harding Lawson Associates (HLA) conducted a soil sampling investigation of the Phase I, II, and III areas. HLA collected surface and subsurface soil samples from 20 soil borings. Soil samples were analyzed for PCBs, cyanide, pesticides, phenols, TPH, sulfides, metals, and VOCs. VOCs were not detected above the laboratory reporting limits in any of the samples collected during this investigation. In September 1989, Romic received EPA approval that the Phase I area was adequately remediated.

- **OCTOBER 1989 EMC CON SOIL INVESTIGATION**

Emcon Associates (Emcon) conducted a soil sampling investigation of the Phase II area, collecting surface and subsurface soil samples from 28 soil borings. Soil samples were analyzed for PCBs, cyanide, pesticides, phenols, TPH, sulfides, metals, and VOCs. VOCs were not detected above the laboratory reporting limits in any of the samples collected during this investigation (Emcon, February 1990).

- **JULY 1990 EMC CON SOIL INVESTIGATION**

Emcon conducted a soil sampling investigation of the Phase III area collecting surface and subsurface soil samples from 23 soil borings. Soil samples were analyzed for PCBs, cyanide, pesticides, phenols, TPH, sulfides, metals, and VOCs. Analytical results indicated VOC concentrations (primarily PCE) above the laboratory reporting limits in samples collected from

17 of the 23 borings. (Emcon, September 1990).

- **APRIL 1991 AND SEPTEMBER 1991 EMCN SOIL INVESTIGATIONS**

In April 1991, Emcon collected surface and subsurface soil samples from 7 sampling locations not sampled during the previous Phase II sampling activities. PCE was detected in sample S-23-1 at a concentration of 0.08 mg/kg (Emcon, June 1991). Split sampling at these 7 sampling locations was conducted by EPA in June 1991. In September 1991, Emcon collected soil samples from one boring (S-24) not sampled during the previous Phase III sampling activities. PCE was detected in samples collected from boring S-24 at concentrations of 4.5 mg/kg at 1 foot bgs and 2.2 mg/kg at 5 feet bgs (Emcon, July 1993).

- **JUNE 1991 THROUGH AUGUST 1992 SOIL EXCAVATION AND LINER INSTALLATION**

Romic conducted initial soil excavation activities at the facility to remove previously detected TPH impacted soils. After unsuccessful attempts to fully remediate the soils in the lateral direction utilizing the initial approach, Romic proposed to remediate the soils in an overlapping grid approach (Emcon, July 1993). The soil excavation and remediation activities were initiated July 1991 and completed in August 1992. Following soil excavation activities, a flexible high density polyethylene liner was installed beneath several of the concrete structures during construction, including each of the containment areas. The liner was installed approximately four to five inches beneath the concrete, with approximately one to two inches of pea gravel atop the liner and native soil beneath the liner (Iris, 2008).

- **2007 WELL INSTALLATION AND SAMPLING**

Drilling and installation activities for the Romic monitor wells were conducted in July 2007. A total of seven groundwater monitor wells, designated RE101 through RE107, were constructed as fenceline monitoring wells in support of the RCRA Part B permit application. No VOCs or semi-volatile organic compounds (SVOCs) were detected in the soils samples collected as part

of the monitor well installation.

The well casing and screen for the wells consisted of schedule 40 polyvinylchloride (PVC), 4-inch diameter well screen with 0.020-inch horizontal slots installed from 65 feet to 100 feet bgs. Following the well completion activities, the monitor wells were developed by swabbing and bailing and then pumping the wells with a submersible pump.

Two rounds of preliminary groundwater quality sampling were conducted at wells RE101 through RE107. Groundwater sampling was conducted at five-foot intervals using the low flow sampling technique as set forth in the EPA approved SAP (Clear Creek Associates, 2007). Groundwater samples collected from monitor wells were analyzed VOCs by EPA Method 8260B, SVOCs by EPA Method 8270C, and the RCRA metals by EPA Methods 6020, 6010B (selenium) and 7470A (mercury). Field parameters were monitored during sample collection. Additionally, one profile sampling interval per well was analyzed for general chemistry parameters during the initial sampling effort.

- **APRIL 2008 SOIL GAS SURVEY**

Drilling and installation of temporary soil gas sampling implants were conducted in April 2008. Each boring was completed as either a single or pair of nested sampling implants by advancing drill rods and/or augers to the total target depth (approximately 10.5 feet or 15.5 feet bgs). Dual-depth soil gas sampling implants were installed at 29 locations and single-depth soil gas sampling implants were installed at 12 locations. Results from the April 2008 soil gas investigation indicated VOC concentrations in 68 of the 70 samples collected, notably PCE and TCE.

Additionally, drilling and installation of a soil vapor extraction test well SVE-01 was conducted in April 2008. The well casing and screen for the well consisted of schedule 40 PVC, 2-inch diameter well screen with 0.020-inch horizontal slots installed from 10 feet to 25 feet bgs. Schedule 40 PVC, 4-inch diameter, blank well casing was installed from just below the land

surface to 10 feet bgs. After the screen and casing were installed, 3/8-inch mesh pea-gravel was installed from the bottom of the borehole (25 feet bgs) to 8 feet bgs, followed by bentonite pellets from 7 feet to 8 feet bgs and granulated bentonite from 6 feet to 7 feet bgs.

- **NOVEMBER AND DECEMBER 2008 DEEP BORING PROGRAM**

LFR, Inc. (now ARCADIS) drilled and installed six nested SVE wells in November and December 2008. Depth-specific soil vapor, soil, and groundwater samples were collected during drilling activities. Depth-specific groundwater samples were collected from the perched zone from between 40 to 60 feet bgs and from the regional aquifer. Two nested, 2-inch diameter PVC SVE wells were installed in each boring. The SVE wells were constructed with Schedule 40 PVC pipe. The SVE well pairs were screened from 10 to 25 feet bgs and 30 to 40 feet bgs with 0.2-inch slotted Schedule 40 PVC. Nested well pairs were installed in borings SVE-02 through SVE-07.

Soil vapor samples collected at selected depths between 15 and 47 feet bgs indicate the presence of vapor-phase VOCs in the vadose zone down to the perched zone. No VOCs were detected in the soil media samples at concentrations above laboratory reporting limits. Groundwater samples were collected from the perched zone between 40 and 60 feet bgs and from the regional aquifer below 70 feet bgs. Analytical results from the perched zone water samples indicated the presence of chlorinated VOCs and acetone. As previously discussed in past reports, VOCs in addition to TCE and PCE were detected in the vapor samples from the soil vapor extraction wells and in the subsurface vapor grab samples; however, these additional compounds are not detected in groundwater samples collected from the regional aquifer during this phase of investigation. Chlorinated VOCs and acetone were also detected in the grab groundwater samples collected at the water table level of the regional aquifer. No VOCs were detected in grab groundwater samples collected at depth (below 165 feet bgs) in the regional aquifer (LFR, 2009a). The investigation results were interpreted to indicate the chemical concentrations and distribution in water samples from the perched zone were

substantially different from the regional aquifer and thus little to no communication was occurring between the two water bearing zones.

- **AUGUST 2009 MONITOR WELL INSTALLATIONS**

Two additional groundwater wells were installed in August 2009 by CCA to provide additional groundwater characterization upgradient and downgradient of the former Romic facility. Preliminary groundwater quality sampling was conducted at depth discrete intervals using the low flow sampling technique noted above. Groundwater samples collected from the monitor wells were analyzed VOCs by EPA Method 8260B. The results of the vertical profiling for VOCs indicated that no VOCs were detected above the laboratory reporting limit in either well.

- **2009 CLOSURE DEMOLITION**

In November 2007, Romic ceased operations at the facility after EPA determined it would not grant a RCRA Part B permit for the facility. Since then, Romic has performed equipment decommissioning, decontamination and removal operations at the facility. The equipment associated with the operation of the SWMUs has been removed including many of the storage units and processing units. Romic contractors performed an EPA-approved RCRA closure of eight hazardous waste management units (HWMUs) and one solid waste management unit (SWMU). The work was completed in 2009 and EPA approval of the closure was issued in March 2010. Closure work included the cleaning and removal of all permitted tanks, process equipment, and piping associated with the facility's permitted activities. The concrete containments were pressure washed and the rinsate tested for compliance. Documentation of the closure work is presented in the *2009 RCRA Closure Report* (Metro Environmental, 2009).

- **2009 RCRA CLOSURE INVESTIGATION**

ARCADIS conducted soil and groundwater sampling in support of RCRA closure activities at the Former Romic Facility between March 30 and April 21, 2009. Eight HWMUs and one SWMU were identified in the Revised RCRA Facility Assessment Report (Booz, Allen, Hamilton, 2004) to

be closed. Thirty two (32) shallow borings were drilled with to a depth of approximately 10 feet bgs and ten deep borings were drilled to a depth of approximately 80 feet bgs (one was drilled to 70 feet bgs).

Soil samples were collected at selected intervals and submitted for laboratory analysis for the RCRA 8 metals by EPA Methods 6010C and 7471B, VOCs by EPA Method 8260B, and SVOCs by EPA Method 8270. Soil samples from the Tank Farm D HWMU were analyzed for pH by EPA Method 9045D and soil samples from the Rail Loading Area SWMU were analyzed for 2,4-dichlorophenoxyacetic acid (2,4-D) by EPA Method 8151A. Groundwater samples were collected from the regional aquifer through a deep boring in each HWMU and SWMU, and submitted for laboratory analysis for RCRA 8 metals (dissolved) by EPA Methods 6010C and 7471B, VOCs by EPA Method 8260B, and SVOCs by EPA Method 8270. Groundwater samples from the Tank Farm D HWMU were analyzed for pH by EPA Method 9040C. Analytical results indicate the presence of metals, VOCs, and SVOCs in soil and groundwater. No 2,4-D was detected in any of the samples analyzed. Lithologic logging of the soil core from the borings confirmed the lithology identified during the 2008 deep soil vapor and groundwater investigation. The perched zone was identified at depths between 40 to 60 feet bgs in each of the deep borings drilled. A more detailed discussion of the RCRA Closure sampling activities and results is included in the *RCRA Closure Report* prepared by Metro Environmental dated July 15, 2009.

- **2009 VADOSE ZONE REMEDIATION ACTIVITIES**

Based on the results of the April 2008 soil vapor survey, a temporary SVE treatment system (SVETS) was installed to reduce the levels of VOCs in the vadose zone and from the perched zone. ARCADIS conducted a SVE pilot test using the temporary system to test VOC mass reduction in the vadose zone soils, and to collect site-specific SVE vacuum response data and vapor recovery rate data to determine if further SVE at the site was appropriate. The pilot test was conducted on December 3, 2008. Data collected during the SVE pilot test indicated that

further SVE was appropriate to reduce VOC mass in the vadose zone soils. Thus, the SVETS used for the pilot test was subsequently operated at the Site from December 4, 2008 through February 20, 2010 for a total of 7,534 hours.

ARCADIS monitored influent and effluent total VOC concentrations on a weekly basis at the SVETS using a portable PID. ARCADIS correlated the influent PID readings to laboratory reported concentration data in order to estimate mass removal. ARCADIS reports that its mass calculations consider the dilution of the field gas to 50% of total flow, based on valve settings on the system. ARCADIS reported approximately 150 pounds of VOCs (hexane equivalent) were removed through February 2010 based on the PID measurements.

ARCADIS collected vapor samples from the SVE wells in December 2008 (Baseline), March 2009 (Rebound), June 2009 (Rebound), and August 2010 (Rebound) and submitted the samples for laboratory analysis for VOCs by EPA Method TO-15. PCE, TCE, acetone and other VOCs were detected in the vapor samples collected from the SVE wells. Further discussion of the effectiveness of the SVE system is included in the Soil Vapor Extraction and Treatment System Operation Report prepared by ARCADIS (ARCADIS, 2011).

- **HISTORICAL INVESTIGATIONS SUMMARY**

Historical operations at the former Romic facility included the storage, handling and treatment of hazardous substances including VOCs. Past practices resulted in the release of VOCs into the subsurface at the former Romic facility. Through infiltration and/or vapor phase migration, VOCs impacted the vadose zone and a perched zone above the regional water table beneath the former Romic facility. As discussed above, data indicate that there is little to no communication between the perched zone and the regional aquifer. Although the overall mass of VOCs released to the subsurface environment is considered to be relatively small, a sufficient mass of both TCE and PCE were released to impact the regional aquifer beneath the southern portion of the Romic facility. TCE and PCE impacted groundwater in the vicinity of the former Romic facility is limited to a very thin zone (approximately 5 to 15 feet thick in most monitoring

locations) near the uppermost water table of the regional aquifer. The narrow width of the plume indicates that the footprint of the release area is relatively small and that the regional groundwater flow direction has remained stable historically over time. The historical source of TCE and PCE impacts has apparently been removed or controlled through on-site remedial actions or through natural attenuation. Additional information regarding the previous investigations and upgrades at the facility is presented in Clear Creek's August 2008 *Current Conditions Report* and Clear Creek's May 2011 *Conceptual Site Model Report*.

3.2 CEMICALS OF CONCERN SUMMARY

A number of chemicals have been detected in groundwater and deep soil samples collected during field investigations at the former Romic facility and as part of the North Central Groundwater Plume investigation. Based on the investigation work conducted to date, the primary chemicals of concern (COCs) for the northern plume in the regional aquifer are PCE, TCE, and 1,1-dichloroethene (1,1-DCE). In addition to being present in the regional aquifer, these compounds have also been detected in deep soil samples, soil vapor samples, and groundwater samples collected from the former Romic facility. The principal source locations for the PCE and TCE contamination appear to be related to historical Southwest Solvents truck unloading and drum storage areas in the south-central portion of the facility (Iris Environmental, 2008).

The vadose zone has been extensively investigated at the former Romic facility. The soil and soil vapor data support the conclusion that there is no discrete, undiscovered source of contamination in the vadose zone at the facility. The relatively small area of impacts, the low concentrations, and the limited vertical distribution of VOCs observed in the environment suggest smaller scale releases – potentially small spills from the unloading and handling of drums or leaks of residual fluids from empty drums in the drum storage area prior to site reconstruction in 1989-1991.

Although the volume of historical releases of TCE and PCE at the former Romic facility may have been limited, they were sufficient to impact the upper few feet of the regional aquifer. On site, groundwater impacts are limited to the southernmost extent of the former Romic facility. The available data indicate that Southwest Solvent's former truck unloading and drum storage areas are the most likely source location for the observed groundwater contamination. Minor contributions may also be attributable to the former railroad unloading area. The distribution of soil vapor on and around the former Romic facility prior to removal by the SVE system suggested an older release that has had time to reach equilibrium in the environment. Multiple lines of analysis support the conclusion that the most recent VOC release to the subsurface occurred prior to 1988.

4.0 POTENTIAL RECEPTOR IDENTIFICATION

This section presents a brief overview of the evaluation of potential receptors that was performed in the Conceptual Site Model Report. Potential pathways for exposure to human receptors were considered to assess whether chemicals associated with the former Romic facility and the northern plume had the potential to contribute to excessive risk for the human population. As previously discussed, COCs are present at low and decreasing concentrations in deep soil vapor and groundwater beneath the former Romic facility, and in shallow groundwater downgradient. Unless a pathway is present and completed between the source and the human receptor, a risk is not present for that population. Populations considered included potential future workers on the former Romic facility and current and future workers employed at the various businesses that overlay the northern plume foot print. There are currently no employees located at the former Romic facility. No specific analysis of risk levels was performed for these potential populations at this time. Only the feasibility of a completed pathway was considered. Additional discussion of the potential exposure pathways at the former Romic facility and over the North Central Plume is included in the Conceptual Site Model report (Clear Creek Associates, 2011a).

4.1 FORMER ROMIC FACILITY

The following potential pathways were considered for the former Romic facility: vapor inhalation, soil ingestion, dermal exposure, and groundwater ingestion or dermal exposure. The former Romic facility is primarily surfaced with concrete and slab-on-grade structures. The limited areas that are unpaved are alternatively surfaced with a gravel blanket or landscaping. The property is not currently occupied. Even if occupied by future workers, a dermal exposure pathway would likely not be completed due to the surface coverings and the absence of COCs in shallow soil. Extensive soil sampling in the permitted unit areas did not detect chemicals present at levels that would cause excessive risk from direct dermal exposure or secondary dust inhalation.

The perched zone continues to represent a potential source of vapor phase VOCs due to diffusion into vadose zone sediments beneath the former Romic facility. Low levels of TCE, PCE and 1,1-DCE were observed in the rebound samples of the SVE wells after nine months of rebound. Rebound was primarily in the deep SVE wells. To gain a perspective on the potential for elevated human health risk from vapor phase VOCs, the rebound concentrations in the shallow SVE wells were compared to the default industrial indoor air risk screening level tables (RSLs) using a conservative attenuation factor derived from the California DTSC guidance (California EPA, 2005). TCE and 1,1-DCE are present in the rebound samples at concentrations below the industrial indoor air default screening levels. In all shallow SVE wells except SVE-3S, the PCE concentrations are below the default industrial indoor air screening level. Therefore, depending on the future development of the former Romic facility, the indoor air pathway is considered to be a potential future exposure pathway that should be considered further in the location of SVE-3S. Currently, however, no buildings are present in the vicinity of well SVE-3S, and thus no completed pathway exists for vapor intrusion at levels of concern for the former Romic Facility. A tier two level of screening using site-specific criteria is proposed to develop site specific risk criteria.

Potable water for drinking, bathing, commercial uses, and similar other uses is supplied by the Lone Butte Industrial Development Corporation (LBIDC) water system. Wells screened in the shallow first water bearing zone have not been located or reported in the area where the northern plume is identified. Therefore, no pathway is competed for groundwater ingestion or use. In addition, the LBIDC and GRIC DEQ have stated that they would enforce a control that prevents installation of shallow drinking water wells in the future.

4.2 NORTH CENTRAL PLUME AREA

The potential exposure pathways considered for the downgradient, offsite portion of the northern groundwater plume were groundwater ingestion, groundwater dermal exposure, and vapor inhalation. No domestic or public water supply wells are impacted by the North Central plume thus there is no completed exposure pathway for ingestion, inhalation or dermal exposure (e.g., bathing). While COCs were detected in grab samples from the discharge of two agricultural supply wells, the VOC concentrations were below maximum contaminant levels (MCLs) for drinking water and significantly below established risk levels for agricultural irrigation. Vapor emissions from the currently decreasing concentrations of VOCs in the northern plume are not considered a completed pathway due to the depth to groundwater and the low concentrations observed at the water table in most locations.

5.0 CONTINUED WATER LEVEL MEASUREMENT AND WATER QUALITY SAMPLING

Collection of regular water level and chemistry data from the regional aquifer is considered critical to completing the RFI and corrective action program. Romic will request either well access or cooperation from GRIC-DEQ to continue semi-annual monitoring of the northern plume. The two components of the monitoring program are discussed below. In addition, Romic is proposing to conduct multi-depth sampling in several Romic and GRIC-DEQ wells to update the characterization of vertical VOC distribution. Groundwater monitoring conducted to date has been conducted pursuant to the EPA approved 2007 and 2009 SAPs (Clear Creek Associates, 2007 and Clear Creek Associates 2009b). Continued groundwater monitoring will be conducted in accordance with the updated SAP included as Appendix A to this RFI Work Plan.

5.1 WATER LEVEL MEASUREMENT

Water levels will continue to be measured on a semi-annual basis utilizing the procedures set forth in the SAP (Appendix A).

5.2 GROUNDWATER MONITORING WELL SAMPLING PROCEDURE MODIFICATIONS

Romic is proposing to change the groundwater sampling method for permanent groundwater monitoring wells from low-flow sampling with dedicated pumps to sampling with passive diffusion bags (PDBs). By modifying the sample collection procedures, the investigation protocol utilized in the north plume will conform with investigations being conducted by GRIC-DEQ representatives. A PDB sampler is a low-density polyethylene bag filled with laboratory grade reagent free water that can be placed in a well. Dissolved VOCs in the groundwater permeate the membrane into the reagent free water contained within the PDB. The bag is left in the well to allow the chemistry of the water in the bag to equilibrate with the water in the well. The PDB bag is then removed from the well and transferred to the appropriate sample container for analysis. Multiple depth sampling is readily performed using PDBs. Procedures

for PDB sampling are detailed in the SAP (Appendix A).

Vertical profiling will consist of sampling with PDBs deployed at approximate five-foot intervals within the saturated portion of each well's screened zone, based on the current groundwater depth. PDB sampling procedures are detailed in the SAP (Appendix A) and the estimated sampling intervals are set forth below:

Well Name	Screened Interval (ft bgs)	Estimated depth to Water (ft below top of casing*)	Sampling Intervals (ft below top of casing*)
RE101	65 – 100	69	82, 87, 92, 97, 102
RE103	65 – 100	64	71, 77, 82, 87, 92, 97
RE104	65 – 100	65	77, 82, 87, 92, 97
RE107	65 – 100	64	71, 77, 82, 87, 92, 97
LB-4	50 – 100	62	68, 74, 80, 86, 92, 98
LB-5	50 – 100	60	70, 76, 82, 88, 94, 98
LB-10	55 – 120	59	100, 106, 112, 118
LB-11	60 – 120	61	100, 106, 112, 118
HC-9	73 – 124	65	103, 113, 118, 123

* RE101 has approximate 3-foot riser

Following the vertical profiling event, a single sampling depth interval will be selected for subsequent sampling events for each well and provided to EPA for approval. Continued semi-annual water quality sampling in the RE series wells for VOCs will be conducted using PDB sampling techniques consistent with GRIC DEQ methods and methods set forth in the SAP (Appendix A).

Groundwater samples will be collected in clean, pre-preserved sampling containers provided by the analytical laboratory, Environmental Science Corporation (ESC), a state certified analytical laboratory.

5.3 QUALITY ASSURANCE/QUALITY CONTROL

One trip blank and one temperature blank will accompany groundwater samples within coolers containing samples for VOC analysis. These blanks will remain unopened until analyzed with the field samples. Duplicate samples will be collected at a 10% occurrence from a randomly chosen well during the sampling events. Data quality objectives and quality control procedures for the groundwater sampling activities are included in the Quality Assurance Project Plan (QAPP) included as Appendix C to this RFI Work Plan.

5.4 GROUNDWATER ANALYSIS

Groundwater samples will be analyzed for VOCs by ESC using EPA Method 8260. Groundwater samples for routine groundwater monitoring activities, including the vertical profiling discussed above, will be submitted for routine turnaround time. Grab groundwater samples collected during the groundwater investigation activities discussed in Section 6 will be submitted for expedited analysis.

6.0 GROUNDWATER INVESTIGATION ACTIVITIES

As discussed in Section 1.1, certain groundwater data gaps were identified in consultation with EPA and GRIC-DEQ. The core questions for the groundwater data gaps was whether the northern VOC plume extends deeper than the existing monitoring network, extends north of well HC-09, and is adequately defined on its distal (western) end. To address these identified data gaps, Romic proposes to drill exploratory borings and collect depth discrete groundwater samples using temporary and/or permanent wells to complete the RFI program. As discussed in the following sections, the proposed investigation activities include:

- Drill and sample three groundwater borings in a north-south transect west of I-10.
- Possible conversion of one or more transect borings into a monitor well.
- Drill and sample one supplemental groundwater boring east of -10 between LB-4 and LB-5.

The general locations of the investigation borings are shown on Figure 2. Final locations may be adjusted based on access considerations. The steps necessary to implement this work are summarized in the following subsections.

6.1 ACCESS AND PERMITTING

Upon EPA approval of the RFI Work Plan, Clear Creek will coordinate with Romic and request access and authorization to install the proposed boring/wells east of I-10 from the Lone Butte Industrial Development Corporation (LBIDC). Clear Creek will prepare and file Notice of Intent to Drill a Groundwater Well information with the GRIC-DEQ for the borings/wells in the vicinity of the former Romic facility. Additionally, an access agreement will be obtained from the Wild Horse Pass development authority for the installation of test boring and potential wells west of I-10 as well as routine groundwater monitoring activities west of I-10. Approval to access the LB and HC series wells will need to be granted by GRIC-DEQ.

6.2 BOREHOLE DRILLING

The borings will be advanced using either rotosonic or dual wall casing advance drilling techniques. Borehole drilling procedures are detailed in the SAP (Appendix A).

6.2.1 Transect West of Interstate 10

A transect consisting of three borings will be installed along the axis of a line running roughly north-south parallel to the west side of I-10 between well LB-14 and a point slightly northwest of LB-2 (Figure 2). The exact locations of the temporary borings will be dependent on access around the new casino property. The purpose of this transect is to evaluate the vertical extent of the plume in the area and to further refine the delineation of the northern boundary of the plume. The borings will be advanced to 140 feet bgs to support vertical groundwater sampling through the plume using the grab groundwater sampling procedures described in Section 6.3, below. Samples will be collected at approximate 10 foot intervals from 90 feet bgs to 140 feet bgs. Sample depths and intervals may be adjusted in the field based on the subsurface conditions encountered.

If PCE concentrations in excess of 5 ug/L are detected in grab groundwater samples collected from the northern boring, then that location will be converted into a permanent monitoring well subject to access approval from the appropriate GRIC entities. If the vertical profile sampling from any of the locations indicates that the bottom of the plume extends deeper than 120 feet bgs, then a new deeper monitoring well will be installed adjacent to existing well LB-10. If it is determined that a new monitoring well is necessary, it will be constructed in accordance with the general procedures set forth in Section 6.5, below. Otherwise, the temporary borings will be abandoned in accordance with GRIC-DEQ well abandonment requirements.

6.2.2 Supplemental Boring

One boring will be drilled east of I-10 along an axis running between LB-4 and LB-5 (Figure 2). The borings will be advanced to 140 feet bgs to support vertical sampling through the plume. Samples will be collected at approximate 10 feet intervals from 90 feet bgs to 140 feet bgs. Sample depths and intervals may be adjusted in the field based on the subsurface conditions encountered.

If, based on vertical profile results, it is determined that the base of the plume extends below the existing monitor well network depth, then the supplemental boring location will be converted into a permanent monitoring well using the procedures described in Section 6.5, below. Otherwise, the temporary boring will be abandoned in accordance with GRIC-DEQ well abandonment requirements.

6.2.3 Vapor Risk Screening Boring

Multiple probe and well-based vapor sampling events have been conducted on and around the former Romic Facility. Sampling performed after completion of the interim corrective action, SVE program, indicates that slightly elevated soil vapor concentrations of VOCs exist in the subsurface. Romic proposes to assess the level of risk associated with the soil vapor for current and future indoor exposure scenarios. To facilitate the assessment, one shallow soil boring will be drilled and sampled at a representative location on site to collect soil samples for geotechnical testing using ASTM procedures. Samples will be collected for analysis for soil bulk density (ASTM D2937), grain density (ASTM D854), soil moisture content (ASTM D2216) and particle size analysis (ASTM D422).

6.3 GRAB GROUNDWATER SAMPLING

Grab groundwater samples will be collected from each of the investigation borings described above using either Hydropunch type or temporary well sample collection techniques. Multiple, vertically spaced groundwater samples will be collected from each boring. Grab groundwater

sampling procedures are detailed in the SAP (Appendix A). Following groundwater sample collection activities, the Hydropunch type or temporary well equipment will be removed from the borehole and the borehole will be properly abandoned in accordance with GRIC-DEQ requirements.

6.4 QUALITY ASSURANCE/QUALITY CONTROL

One trip blank and one temperature blank will accompany groundwater samples within coolers containing samples for VOC analysis. These blanks will remain unopened until analyzed with the field samples. Duplicate samples will be collected at a 10% occurrence during the sampling events. Specific quality control (QC) procedures are detailed in the QAPP (Appendix B).

6.5 MONITOR WELL INSTALLATION

If the results of the grab groundwater sampling are interpreted to justify installing a new monitoring well, following accelerated consultation with EPA and GRIC-DEQ, then the new monitor well will be installed in accordance with the general procedures described in this section. The preliminary design of the well is shown on Figure 3. The top twenty feet of the wells will be constructed of new 4-inch diameter low carbon steel casing followed by new flush threaded Schedule 40 PVC blank casing. The well screen will be constructed with approximately 40 feet of 4-inch flush threaded Schedule 40 PVC well screen with 0.020-inch factory cut slots and equipped with a Schedule 40 bottom cap. The exact length and depth of the well screen will be based on the results of the grab groundwater sampling described above.

A filter pack of 10-20 sieve size silica sand will be installed across the screen zone and extend approximately 10 feet above the screen zone to prevent the penetration of the seal material into the well. A 10 foot bentonite seal will be placed above the filter pack using bentonite chips and hydrated with potable water. A slurry of bentonite grout will be emplaced above the bentonite seal up to 20 feet bgs and a cement seal will be placed from 20 feet bgs to ground surface. The well will be completed at the surface in a flush-mounted traffic-rated well vault and secured with a locking waterproof seal at the well head. The well will be surveyed to

provide location and elevation information to the nearest 0.01 foot consistent with GRIC datum.

After well installation, the well will be developed by using swabbing and air lifting techniques. The well will be surged in an effort to loosen and remove fine sediments from the filter pack. After surging, sediments brought into the well will be removed by bailing. All water generated by the well installation and development will be contained onsite either at the former Romic facility or an alternate location provided by GRIC until characterized for disposal.

7.0 REPORTING

Upon completion of boring and well installation and groundwater sampling activities, Clear Creek will prepare an RFI Summary Report documenting the drilling and construction procedures, lithologic conditions, as-built diagrams, well survey results, groundwater elevations, and the results from the soil and groundwater sampling.

In addition, the report will include a summary screening of the preliminary corrective measures described in Section 7. Integrated into the RFI data analysis will be an update to the CSM, if necessary based on the additional data collected, and a reassessment of the vertical and horizontal extent of the northern plume. A possible outcome of the assessment may be the determination of whether installation of a distal compliance monitor well is necessary for regulatory purposes.

8.0 PRELIMINARY CORRECTIVE MEASURES

As required by the AOC, the following potential corrective measures are currently being considered for further assessment. Each will be further considered in light of findings from the RFI program. Romic is also considering combining the RFI implementation with a Corrective Measures Study (CMS) in order to expedite the decision-making process and implementation if a corrective measure is found to be needed. These preliminary corrective measures were identified in recognition that groundwater concentrations have declined significantly in the monitor wells at and downgradient of the facility since facility wells were installed in 2007 (CCA and Arcadis, 2011a).

8.1 MONITORED NATURAL ATTENUATION (MNA)

Natural attenuation may be occurring within all or portions of the northern plume. Given the relatively aerobic conditions of the regional aquifer, it is considered unlikely that the reduction of concentrations observed in the well network is due to biodegradation. Although abiotic destruction of some compounds does occur, it is more likely that nondestructive attenuation mechanisms are responsible, including sorption, dispersion, dilution from recharge, and volatilization (Wiedemeir, et al., 1998). As part of the screening process, parallel lines of evidence will be developed from existing data and data generated during the RFI implementation. Key evidence may include plume stability, chemical trends, plume shape, chemical sorption, and estimation of dilution rates. Recommendations resulting from the analysis may consist of modifications to the current monitoring program to better document MNA of the northern plume. Ultimately, performance monitoring for a MNA remedy would follow the guidelines laid out in the OSWER Directive 9200.4.17 on Monitored Natural Attenuation (1997) or similar guidance.

8.2 LIMITED SOURCE AREA CHEMICAL OXIDATION

Chemical oxidation is being considered in limited areas on the former Romic facility in association with the anticipated drilling effort to abandon the existing SVE wells. Chemical oxidation is being considered as a final step to accelerate the apparent MNA currently occurring in groundwater at the former Romic facility. Cost effective delivery of oxidant solutions is limited by the inability to utilize direct push methods to reach the aquifer. To address this limitation, Romic is considering the feasibility of gaining access to the perched zone and regional aquifer by extending an injection lance through selected onsite SVE wells prior to SVE well abandonment. As part of the RFI process, the optimal chemical oxidation chemistry and physical challenges to this possible corrective measure will be screened for feasibility.

8.3 COMBINATION MNA/SOURCE AREA CHEMICAL OXIDATION

This combination approach would be considered if MNA was identified as an appropriate corrective measure for the northern plume and limited, final, source area VOC reduction is deemed appropriate and feasible.

9.0 PROJECT MANAGEMENT

A project organizational chart is presented on Figure 4. Brief descriptions of the specific roles and responsibilities for key team members are discussed below.

Client Representative / Project Coordinator: Mr. Chris Alger of Iris Environmental, is the designated Client Representative and is the Project Coordinator responsible for implementation of all tasks required by the Consent Order. His responsibilities include defining project objectives, establishing the project team, and securing project funding from Romic Environmental. Mr. Alger also has responsibility for communicating with the EPA Project Manager, Mr. John Moody.

RFI Project Manager: Mr. Thomas R. Suriano, R.G., of Clear Creek Associates will be the Project Manager responsible for the implementation of the RFI tasks identified in this Work Plan. Mr. Suriano reports to Mr. Chris Alger. Mr. Suriano's responsibilities include directing project activities, assigning staff, ensuring project schedules are met, preparing documents, and ensuring that the work meets the Consent Order requirements.

Field Supervisor: Mr. Geno Mammini, R.G., of Clear Creek Associates will serve as field supervisor. The Field Coordinator reports to the RFI Project Manager. The Field Supervisor has responsibility for implementing and/or overseeing project staff in implementing the field work and ensuring that the requirements of this RFI Work Plan are met.

10.0 SCHEDULE

A generalized schedule for implementation of the work is presented in Figure 5.

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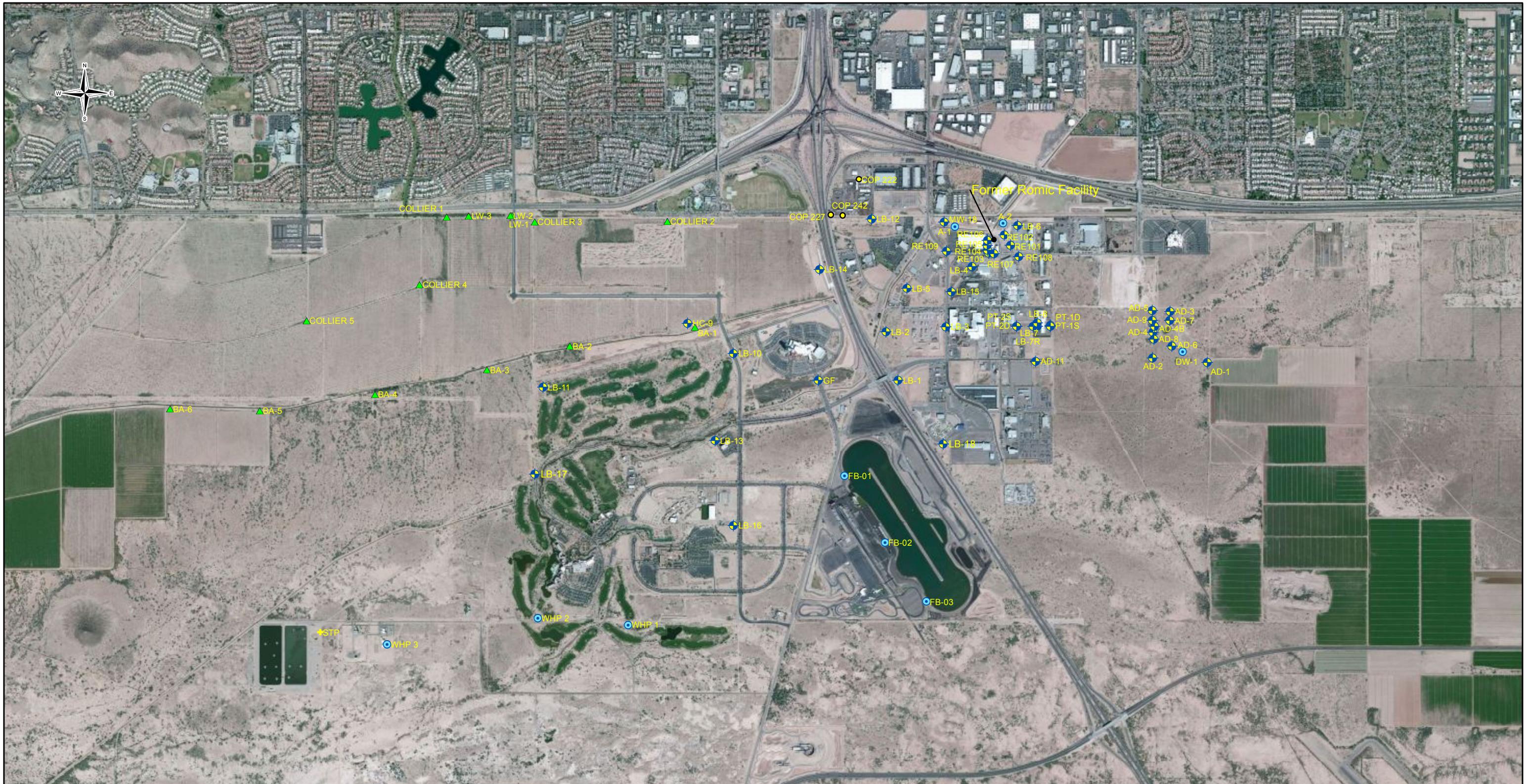
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FIGURES



RCRA Facility Investigation (RFI) Work Plan
Former Romic Environmental Technologies Facility
Gila River Indian Community, Arizona

December 2011
212001



VICINITY / WELL LOCATION MAP

Explanation

- Monitor Well
- ▲ Agricultural Well
- Drinking Water Well
- COP
- POC Well

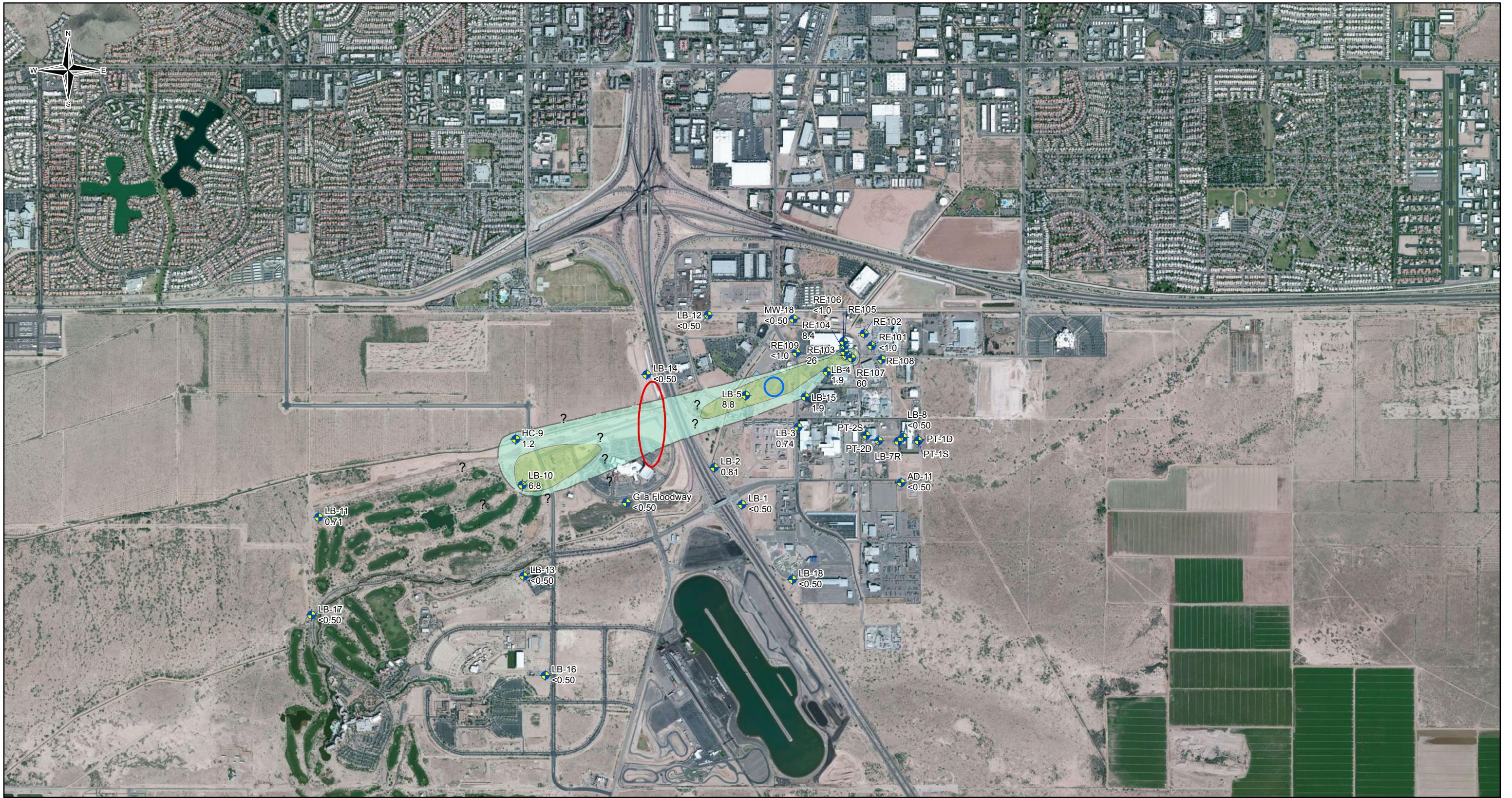
0 0.25 0.5 1 1.5 Miles

6155 East Indian School Road
Suite 200
Scottsdale, Arizona 85251
(480) 659-7131

CLEAR CREEK ASSOCIATES

RCRA Facility Investigation (RFI) Work Plan
Former Romic Environmental Technologies Facility

Figure 1
December 2011



Explanation

◆ August 2011 PCE Concentration (ug/L)

PCE Concentration Contours

<1.0 ug/L
>1.0 ug/L
>5.0 ug/L
>25 ug/L
>50 ug/L

○ Proposed Transect Location

○ Proposed Boring Location

Areas of Additional Investigation

0 0.25 0.5 Miles

World Imagery

0 0.25 0.5 Miles

6155 East Indian School Road
Suite 200
Scottsdale, Arizona 85251
(480) 659-7131

CLEAR CREEK ASSOCIATES

RCRA Facility Investigation (RFI) Work Plan
Former Romic Environmental Technologies Facility

Figure 2
December 2011

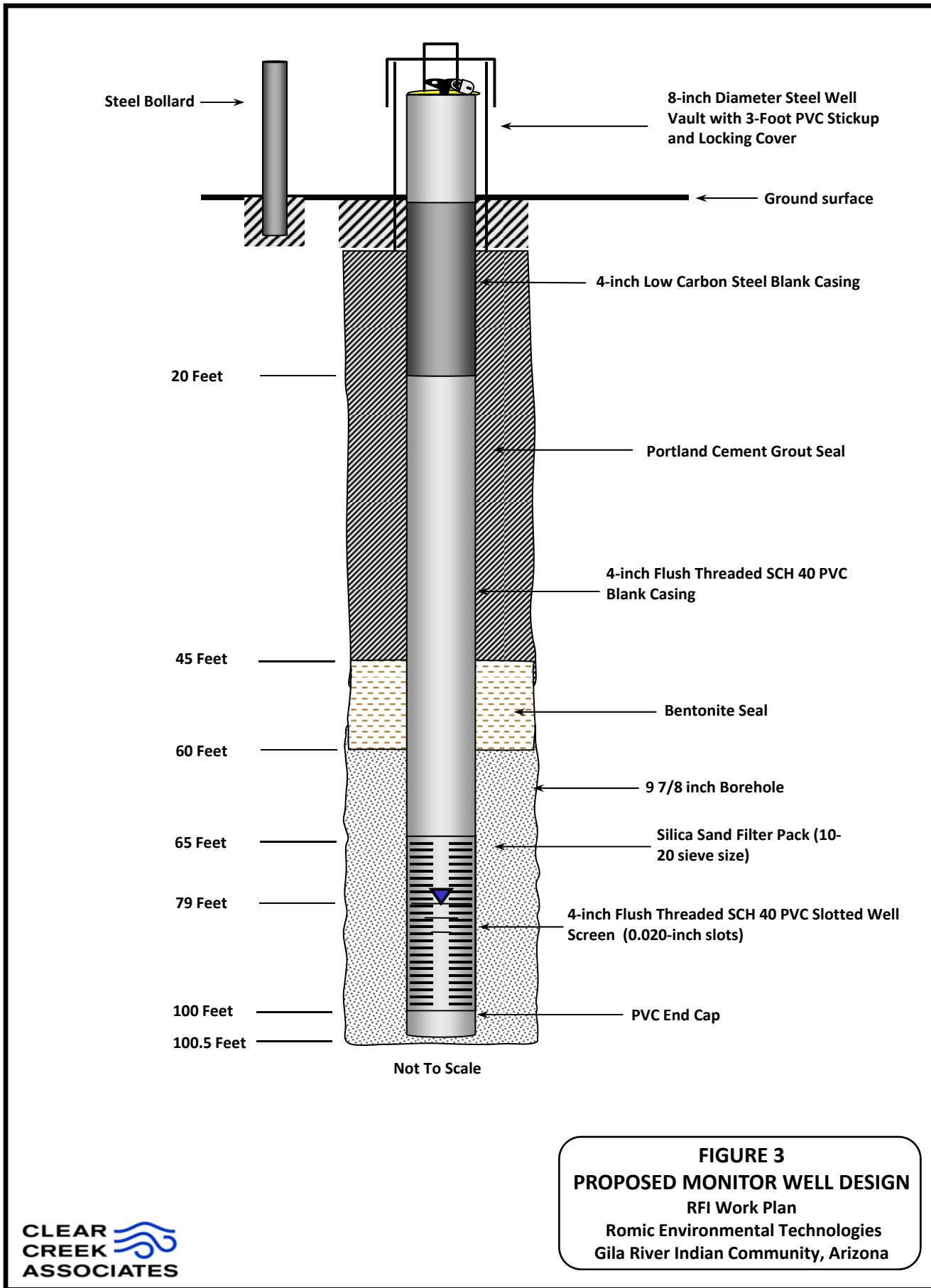


FIGURE 3
PROPOSED MONITOR WELL DESIGN
 RFI Work Plan
 Romic Environmental Technologies
 Gila River Indian Community, Arizona

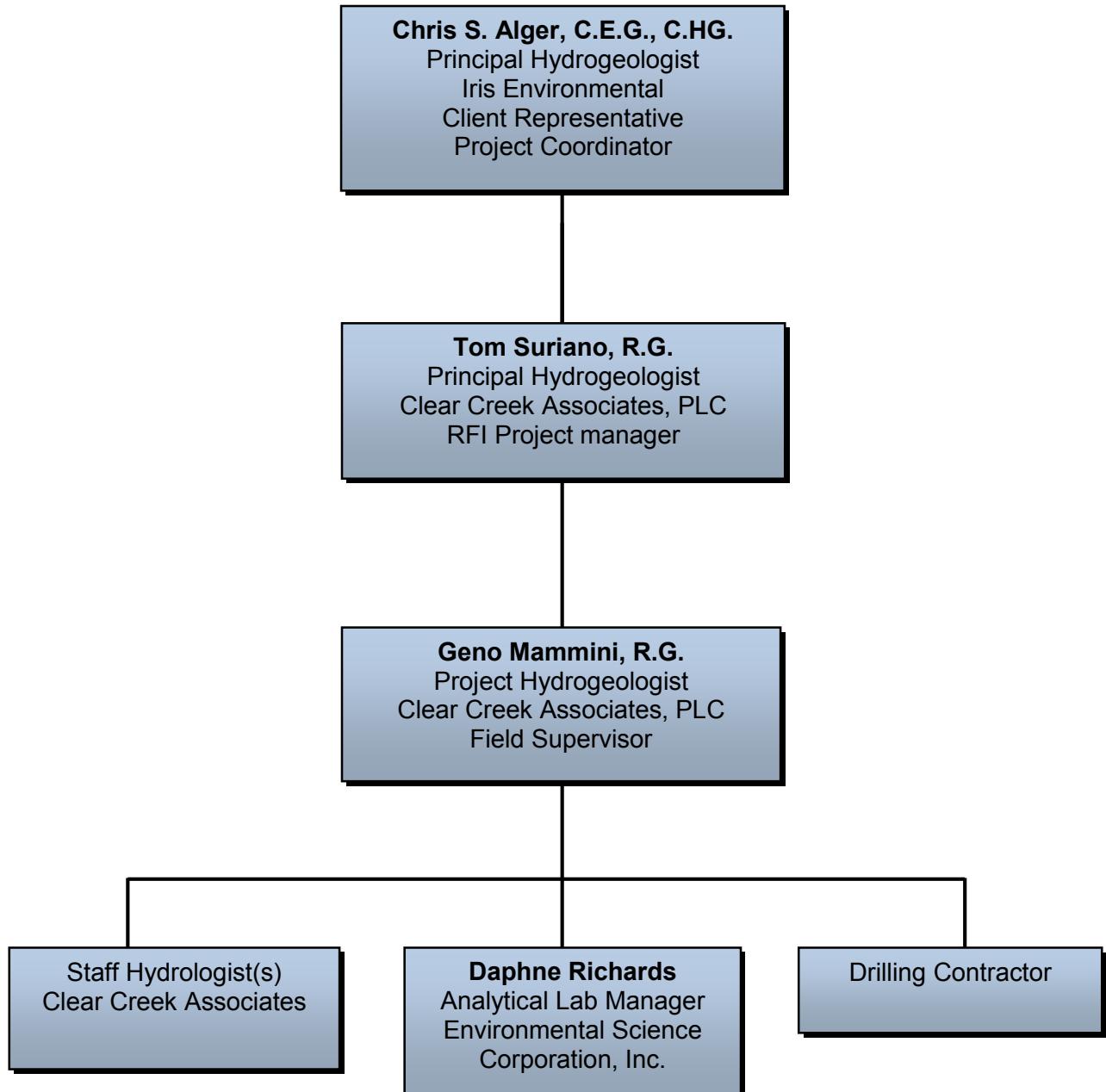


FIGURE 4
PROJECT ORGANIZATIONAL CHART
RFI Work Plan
Romic Environmental Technologies
Gila River Indian Community, Arizona

	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6	Week 7	Week 8	Week 9	Week 10	Week 11	Week 12	Week 13
PDB Installation and Equilibration													
PDB Groundwater (GW) Sampling													
PDB GW Laboratory Analyses													
GW Investigation - Soil Borings													
Grab GW Laboratory Analyses													
Monitor Well Installation (if applicable)													
Initial Well Sampling - VOC Depth Profiling (if applicable)													
Monitor Well GW Laboratory Analyses (if applicable)													
Corrective Measures Study													
Draft RFI Report													

Note: Generalized Project Schedule assumes EPA approval of the RFI Work Plan and the applicable access agreements have been secured.

APPENDIX A

SAMPLING AND ANALYSIS PLAN



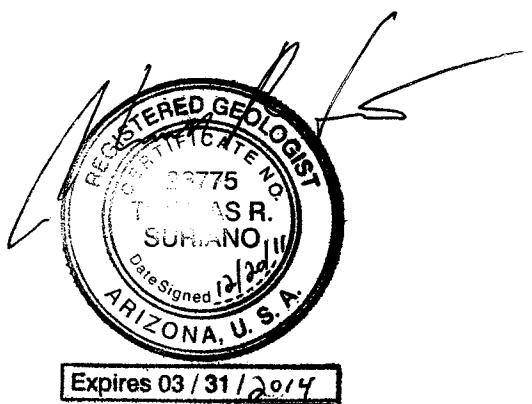
RCRA Facility Investigation (RFI) Work Plan
Former Romic Environmental Technologies Facility
Gila River Indian Community, Arizona

December 2011
212001

RFI WORK PLAN APPENDIX A

SAMPLING AND ANALYSIS PLAN RCRA FACILITY INVESTIGATION AND CONTINUED GROUNDWATER SAMPLING

**FORMER ROMIC ENVIRONMENTAL TECHNOLOGIES FACILITY
LONE BUTTE INDUSTRIAL PARK
GILA RIVER INDIAN COMMUNITY, ARIZONA**



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**December 2011
Project No. 212001**



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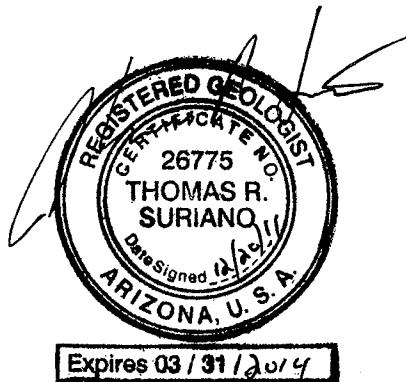
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1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been prepared for the RCRA Facility Investigation (RFI) of the former Romic Environmental Technologies Corp. (Romic) facility located in the Lone Butte Industrial Park in the Gila River Indian Community (GRIC) near Chandler, Arizona (Figure 1). The SAP presents and updates protocols previously forth in Clear Creek Associates' June 2007 *Sampling and Analysis Plan* (Clear Creek, 2007) and June 2009 *Sampling and Analysis Plan* (Clear Creek, 2009). This SAP presents the protocols to be followed during the RFI, which will consist of a series of soil borings and depth discrete groundwater sampling. If necessary, the RFI may include well drilling, monitor well construction and preliminary groundwater sampling of an offsite monitor well in the vicinity of the former Romic facility. Additionally, this SAP updates the groundwater monitoring and sample collection portions of the previous SAPs to include passive diffusive bag (PDB) sampling methods into the groundwater sampling program. The objective of the SAP is to establish the procedures so that the data collected are accurate and representative of field conditions. The procedures presented in this SAP are consistent with industry-standard practices, *ASTM Standards on Groundwater and Vadose Zone Investigations* (ASTM, 1994) and various guidance documents published by the U.S. Environmental Protection Agency (EPA). Section 4.0 (Preliminary Investigation and Source Reduction) of the Gila River Indian Community Department of Environmental Quality (GRIC-DEQ) *Draft Site Characterization and Remedial Action for Releases Which Impact or Threaten Groundwater Quality Guidance Document* (SCRA Guidance) (GRDEQ, 2004) was used as a reference for preparing this SAP for the RFI at the former Romic facility. This SAP has no significant deviations from the contaminant standards or well installation, groundwater sampling methods and procedures set forth in the SCRA Guidance.

This SAP is organized as follows.

- Section 1.0 Introduction, Background and Objectives.
- Section 2.0 Water Level Measurement and Water Quality Sampling.
- Section 3.0 Borehole Construction, Lithologic Logging and Equipment Decontamination.
- Section 4.0 Monitor Well Construction.
- Section 5.0 Documentation.
- Section 6.0 References.

1.1 BACKGROUND

The facility began operations in 1975 as Southwest Solvents and later as Southwest Solvent Industrial Recycling. For purposes of this work plan, these operations are collectively referred to as Southwest Solvents. Primary operations at Southwest Solvents consisted of hazardous waste recycling. While operating as Southwest Solvents, the majority of the site was unpaved, with the exception of the loading dock, the main building and four to five isolated material handling areas (Booz, Allen, Hamilton, November 2004). Following several EPA site inspections, Southwest Solvents was notified of numerous violations including unsatisfactorily operating a facility generating hazardous waste, unsatisfactorily operating a treatment storage or disposal (TSD) facility, and not furnishing information in conjunction with an interim status facility application. Based on these violations, EPA issued a Consent Agreement / Final Order (CA/FO) (RCRA-09-88-0002) to Southwest Solvents to investigate and remediate releases and to dispose of excess waste at their facility. Shortly thereafter in 1988, Romic purchased the facility out of bankruptcy from Southwest Solvents.

Romic operated under interim status since 1988 when they purchased the facility from Southwest Solvents until it shut down in 2007. In January 1999, Romic was acquired by U.S. Liquids, and subsequently, acquired by ERP Environmental Services, Inc. in August 2003. The facility operated under interim status as Romic Environmental Technologies Corporation. In November 2007, Romic ceased operations at the facility after a RCRA Part B permit was not granted for the facility. No hazardous waste treatment and recycling operations are currently being conducted at the facility and no hazardous waste remains at the facility.

1.2 PURPOSE AND OBJECTIVES

As stated in the Administrative Order on Consent (AOC) and EPA guidance, the focus of the RFI is to identify the nature and extent of releases of hazardous waste and/or hazardous constituents at or from the former Romic facility which may pose an unacceptable risk to human health and the environment. To date, Romic has conducted numerous investigations nature and extent of releases at the former facility and implemented interim corrective action measures (ICAM) to mitigate soil impacts, and has participated in TRIAD style investigations both onsite and offsite to further characterize the nature and extent of vapor and groundwater impacts. The results of the investigations and corrective measures are summarized in the RFI Work Plan and documented in detail in the Current Conditions Report (Clear Creek Associates, 2008) and the Conceptual Site

Model (CSM) report (Clear Creek Associates, 2011). As a result of this prior work, this RFI is targeted at resolving remaining investigation data gaps and collecting data considered necessary for assessing the need for additional corrective action.

Continued groundwater monitoring is necessary to support the evaluation of potential interim corrective action measures. Additionally, groundwater data gaps were discussed during the June 21-22, 2011 meetings with EPA and the GRIC-DEQ. The essence of the groundwater data gaps was whether the northern volatile organic compounds (VOC) plume extends deeper than the existing monitoring network, extends north of well HC-09, and is adequately defined on its distal (western) end. The procedures for implementing the work necessary to conduct on-going groundwater monitoring and to address the identified data gaps are set forth in this SAP.

2.0 WATER LEVEL MEASUREMENT AND WATER QUALITY SAMPLING

Collection of regular water level and chemistry data from the regional aquifer is considered critical to completing the RFI and corrective action program. Romic will request either well access or cooperation from GRIC-DEQ to continue semi-annual monitoring of the northern plume. The two components of the monitoring program are discussed below. In addition, Romic is proposing to conduct multi-depth sampling in several Romic and GRIC-DEQ wells to update the characterization of vertical VOC distribution.

2.1 MONITORING SCHEDULE

Water level and water quality sampling will be conducted on a semi-annual basis. Monitoring events will be conducted in March and August to be consistent with the monitoring schedule implemented by GRIC-DEQ.

2.2 WATER LEVEL MEASUREMENT

Static water levels will be measured in each Romic monitoring well (RE-101 through RE-109) using an electric water level sounder with graduated markings in feet and hundredths of a foot. All measurements will be measured from an established measuring point that has been surveyed in order to determine elevation of water above mean sea level. All water level measurements will be measured from the point where the water level indicator registers the maximum sound. Water level measurements will be repeated until a difference of less than 0.1 foot between consecutive measurements is obtained. Depth to water, measurement date and time, and the initials of the sampler/measurer will be annotated on a water sampling form.

2.3 GROUNDWATER SAMPLING PROCEDURES

Groundwater sampling events will be conducted utilizing the PDB sampling method. By modifying the sample collection procedures, the investigation protocol utilized in the north plume will conform with investigations being conducted by GRIC-DEQ representatives. A PDB sampler is a low-density polyethylene bag filled with laboratory grade reagent free water that can be placed in a well. Dissolved VOCs in the groundwater permeate the membrane into the reagent free water contained within the PDB. The bag is left in the well to allow the chemistry of the water in the bag to equilibrate with the water in the well. The PDB bag is then removed

from the well and transferred to the appropriate sample container for analysis. Multiple depth sampling is readily performed using PDBs.

2.3.1 PDB SAMPLER DEPLOYMENT AND RETRIEVAL

New, pre-filled PDB bags will be obtained from the analytical laboratory, Environmental Science Corporation (ESC), a state certified analytical laboratory, prior to the sampling effort. PDB sampling is accomplished by lowering low-density polyethylene bags filled with dionized water into the well and allowing the water in the bag to equilibrate to the water in the well. The depth intervals to position the PDBs will be determined before the sample round and the lines to secure the PDBs will be measured and prepared prior to the sampling event. Pre-filled PDBs are attached to a polyester rope or stainless steel line. A weight is attached to the lowermost bag to counterbalance the buoyancy of the bag(s). The assembly is lowered to the appropriate depth. The line is secured at the well head and the well should be sealed to prevent surface water from entering the well. The PDBs remain undisturbed in the well to equilibrate for approximately one to two weeks. After the equilibration period the PDB samplers are removed by pulling up the attached line. The PDB sampler should be examined for tears, algae or other coating. The condition of the PDB should be noted in the sample collection record. If there are tears in the bag the sample will be rejected. Remove the PDB from the line and remove excess water from the exterior of the PDB to minimize the potential for cross contamination.

2.3.2 GROUNDWATER SAMPLE COLLECTION

Groundwater samples will be collected in clean, pre-preserved sampling containers provided by ESC. All preservatives will be prepared and added to the appropriate sampling container by the laboratory prior to sampling. Sample containers will be kept in a clean, cool, and secure location until immediately prior to use.

The water from the bag can be transferred to the volatile organic analysis (VOA) vials by one of several methods including pouring from the fill plug at the top of the bag, inserting a discharge device or cutting a hole in the bag with decontaminated scissors. The sampling hydrogeologist will wear nitrile gloves while collecting samples. Water from the PDB bags is transferred into three 40-ml glass vials with Teflon lined septa (or VOA vials) will be filled for VOC analysis. The container will be filled to slightly overflowing, forming a convex meniscus at the mouth of the container. The cap will be placed upon the convex meniscus and screwing on tightly to seal the

container. To check that the sample is air free, the container will be inverted gently and the cap gently tapped. The absence of entrapped air indicates a successful seal. When air is encountered in the container, the sample and container will be discarded and the water sample will be recollected.

2.3.3 SAMPLE HANDLING AND CUSTODY

Each groundwater sample will be labeled immediately upon collection including: sample identifier, the date and time of sample collection, initials of the sampler, analysis requested, project number, and client name. The sample identifier consists of the well (or boring) ID and depth interval sampled (e.g. the samples collected from the 88 foot bgs interval from well LB-5 will be identified as LB-5-88). All information entered on the label will exactly match the information contained on the chain of custody document. After groundwater samples are collected, they will be placed in an appropriate container (either of VOA vial holder or resealable plastic bag) and then immediately placed in an ice chest maintained at 4 degrees Celsius using wet ice. A chain of custody will be maintained at all times with the collected samples and turned over to the laboratory when samples are delivered each sampling day. Custody seals are not necessary if the samples do not leave the custody of the sampler prior to delivery to the laboratory. If the samples leave the custody of the sampler for any reason, then one or more of each sample container, resealable bag, or each cooler will be sealed with a transportation security seal (chain-of-custody seal) containing the sampler's initials. The groundwater samples will be submitted on a standard 5-day turnaround time (TAT).

2.3.4 SAMPLE DOCUMENTATION

Sample documentation will consist of the following (examples are in Attachment A):

- Daily Field Reports.
- Sample Labels.
- Chain-of-Custody.

Daily Field Report

A daily field report will be maintained by the field technician collecting the samples. These logs will contain specific records of daily field activities. Field reports will include, but are not limited to, the following:

- Job number.
- Well ID.
- Date.
- Field Technicians (Hydrogeologists).
- All sampling information (i.e. depth to water, date and time PDB bags were deployed, date and time PDB bags were collected, and condition of the PDB).
- Any problems or deviations from scheduled activities.
- Any other pertinent information.
- Signature of the field technician responsible for the sample collection.

A copy of the daily field reports will be submitted to the project office on a daily basis.

Sample Labels

Each groundwater sample will be labeled immediately upon collection including: sample identifier, the date and time of sample collection, initials of the sampler, analysis requested, project number, and client name. The sample identifier consists of the well (or boring) ID and depth interval sampled (e.g. the samples collected from the 88 foot bgs interval from well LB-5 will be identified as LB-5-88). All information entered on the label will exactly match the information contained on the chain of custody document.

Chain-of-Custody

A laboratory Chain-of-Custody will be completed by the field technician collecting the sample and will accompany each sample cooler. Whenever a sample is transferred to another responsible party, the receiving party must sign off on the Chain-of-Custody form. Each Chain-of-Custody form will include the following information:

- Consultant name, address, phone number.

- Project manager.
- Project number.
- Well ID.
- Date: date sample was collected.
- Time: time sample was collected.
- Matrix: type of sample collected (i.e. water, soil).
- Analysis requested: type of analysis to be performed on the sample.
- If preserved or not.
- Number of containers used.
- Relinquished by: signature of the sampler.
- Date/Time relinquished: the date and time the sample was relinquished by the technician.
- Received by: signature of whomever received the sample must sign for it.
- Received from laboratory by: signature of the person at the lab who officially accepted the samples.
- Date/Time: the date and time the sample was received by the laboratory.
- Temperature of samples received.
- Remarks: Final remarks about the samples after acceptance by the laboratory.

A copy of the Chain-of-Custody records will be submitted to the project office on a daily basis as well as returned by the laboratory with the analytical results.

2.4 QUALITY ASSURANCE/QUALITY CONTROL

One trip blank and one temperature blank will accompany groundwater samples within coolers containing samples for VOC analysis. These blanks will remain unopened until analyzed with the field samples. Duplicate samples will be collected at a 10% occurrence from a randomly chosen well during the sampling events.

2.5 VERTICAL PROFILE SAMPLING

2.5.1 EXISTING WELLS

One round of vertical profile sampling will be conducted in the wells noted below utilizing PDB groundwater sampling techniques following EPA approval. Implementation of this task is dependent upon acquiring access to the GRIC DEQ wells for sampling. Vertical profiling will consist of sampling with PDBs deployed at approximate five-foot intervals within the saturated portion of each well's screened zone, based on the current groundwater depth. The estimated sampling intervals are set forth below:

Well Name	Screened Interval (ft bgs)	Estimated depth to Water (ft below top of casing*)	Sampling Intervals (ft below top of casing*)
RE101	65 – 100	69	82, 87, 92, 97, 102
RE103	65 – 100	64	71, 77, 82, 87, 92, 97
RE104	65 – 100	65	77, 82, 87, 92, 97
RE107	65 – 100	64	71, 77, 82, 87, 92, 97
LB-4	50 – 100	62	68, 74, 80, 86, 92, 98
LB-5	50 – 100	60	70, 76, 82, 88, 94, 98
LB-10	55 – 120	59	100, 106, 112, 118
LB-11	60 – 120	61	100, 106, 112, 118
HC-9	73 – 124	65	103, 113, 118, 123

* RE-101 has an approximate 3-foot riser.

Following the vertical profiling event, a single sampling depth interval will be selected for subsequent sampling events for each well and provided to EPA for approval. Continued semi-annual water quality sampling will be conducted using PDB sampling techniques at the selected depth interval.

2.5.2 NEWLY INSTALLED WELLS

In the event any new monitor wells are installed pursuant to section 4.0 of this SAP, one round of vertical profile sampling will be conducted in the wells utilizing PDB groundwater sampling techniques to identify the appropriate sampling interval. Vertical profiling will consist of sampling with PDBs deployed at approximate five-foot intervals within the saturated portion of each well's screened zone, based on the current groundwater depth and the results of the grab groundwater sampling conducted during well installation. The initial vertical depth profiling and future groundwater monitoring will be conducted using the PDB sampling technique. The well will be allowed to equilibrate for at least one-week after development prior to the initial sampling effort. Following the vertical profiling event, a single sampling depth interval will be selected for subsequent sampling events for each well and provided to EPA for approval. Continued semi-annual water quality sampling will be conducted using PDB sampling technique as set forth in Section 2.3 at the selected depth interval.

3.0 BOREHOLE DRILLING, LITHOLOGIC LOGGING AND EQUIPMENT DECONTAMINATION

Boreholes will be advanced into the subsurface and depth discrete groundwater samples will be collected using temporary and/or permanent wells at strategic locations across the northern VOC plume to complete the RFI program. A transect consisting of three borings will be installed along the axis of a line running roughly north-south parallel to the west side of I-10 between well LB-14 and a point slightly northwest of LB-2 (Figure 2). The exact locations of the temporary borings will be dependent on access around the new casino property. The purpose of this transect is to evaluate the vertical extent of the plume in the area and to further refine the delineation of the northern boundary of the plume. One additional boring will be drilled along an axis running between LB-4 and LB-5 (Figure 2) one shallow soil boring will be drilled at the former Romic facility and to assess the level of risk associated with the slightly elevated soil vapor concentrations of VOCs for current and future indoor exposure scenarios. The procedures for advancing the boreholes, conducting lithologic logging of the subsurface sediments and collecting depth discrete groundwater samples are set forth in this Section. The procedures for constructing the well (if necessary) are discussed in Section 5.

3.1 ACCESS AND PERMITTING

Clear Creek will coordinate with Romic and request access and authorization to install the proposed boring/wells from the Lone Butte Industrial Park. Clear Creek will prepare and file Notice of Intent to Drill a Groundwater Well information with the Arizona GRIC-DEQ for the borings/wells in the vicinity of the former Romic facility. Additionally, an access agreement will be obtained from the Wild Horse Pass development authority for the installation of test boring and potential wells west of I-10 as well as routine groundwater monitoring activities west of I-10. Access will also be obtained from the LDIDC for the proposed work east of I-10. Approval to access the LB and HC series wells will need to be granted by GRIC-DEQ and the Wild Horse Pass casino.

3.2 BOREHOLE DRILLING

The borings will be advanced using either rotosonic or dual wall casing advance drilling techniques. All drilling will be performed under the supervision of an experienced

hydrogeologist to ensure adherence to design specifications. A ‘tailgate’ safety meeting will be held at the outset of field operations with drilling personnel to understand the safety requirement for working in the vicinity of the former Romic facility. Arizona Blue Staking Service will be contacted to mark all utilities in the area of the proposed drill sites and Clear Creek will also retain a private locator to identify utility locations at the proposed drill sites. Due to the proximity of the proposed boring locations to utilities, the initial 10 feet of drilling will be conducted by potholing.

Clear Creek will provide continuous inspection services during the drilling, and potentially, well construction period. The borehole diameter of the holes will be approximately 9-7/8 inches. Boreholes will be advanced to an approximate depth of 140 feet bgs. The total drill depth is subject to change based on site conditions observed by the hydrogeologist. Borehole cuttings will be collected at a minimum of 10-foot intervals for lithologic examination by the field geologist as set forth in Section 3.3, below.

Soil samples will not be collected for VOC laboratory analysis. Soil samples will be field screened for a qualitative assessment of the potential presence of contamination using a photo-ionization detector (PID). To screen with the PID, a portion of soils from the end of the sample collection barrel will be placed in a resealable plastic bag. The tip of the PID will be inserted into the airspace above the soils and the readings recorded. To distinguish from positive readings potentially associated with naturally occurring organic matter in the soils, only significant deviations of the PID meter will be identified as potentially contaminated unless other physical indicators (e.g. staining or odors) are also present to support such a determination. Prior to each day's use, the PID will be calibrated according to the manufacturer's calibration procedures (Attachment B).

The borings will be advanced to 140 feet bgs to support vertical groundwater sampling through the plume using the grab groundwater sampling procedures described in Section 4.5, below. If it is determined that a new monitoring well is necessary, it will be constructed in accordance with the general procedures set forth in Section 5.0, below. Otherwise, the temporary borings will be abandoned with Portland Type II Cement slurry with up to 5 percent bentonite added to the cement slurry, in accordance with GRIC-DEQ well abandonment requirements.

3.3 LITHOLOGIC LOGGING

The on-site hydrogeologist will monitor drilling activities and maintain a lithologic log of drill cuttings. Lithologic descriptions will follow the Unified Soil Classification System (USCS) and include the following information:

- Textural classification
- Color
- Sorting
- Roundness
- Consistency or relative density
- Plasticity
- Reaction to dilute hydrochloric acid

The hydrogeologist will take into account any alterations to soil samples caused by the drilling process. Representative cutting samples will be stored in plastic chip tray containers for future reference. Additionally, the hydrogeologist will note field conditions, depth to water encountered during drilling, drill rig behavior, and general site observations.

3.4 DEPTH DISCRETE GRAB GROUNDWATER SAMPLING PROCEDURES

The borings will be advanced to 140 feet bgs to support vertical groundwater sampling through the plume using the grab groundwater sampling procedures. Samples will be collected at approximate 10 feet intervals from 90 feet bgs to 140 feet bgs. Sample depths and intervals may be adjusted in the field based on the subsurface conditions encountered. The depth specific samples will be collected using either a hydropunch type sampler (or similar technique) or temporary mini-wells.

Hydropunch Type Sampling – The following steps will be followed for collecting grab groundwater samplers with a Hydropunch II™ tool or similar technique:

- The drilling subcontractor will advance the borehole to the appropriate depth for collecting samples. The borehole will be stopped approximately 2-feet above the desired grab groundwater sampling interval.
- Static water level will be recorded inside the drill string using a water level sounder.
- The drilling contractor will prepare the Hydropunch II™ tool in accordance with the manufacturer's instructions and lower the device to the bottom of the borehole.
- The sampling device will be driven to the appropriate depth below the bottom of the borehole.
- The drilling subcontractor will retract the drive rod to expose the sampling screen.
- The sampler will be left in place for an appropriate amount of time to fill with water.
- A grab groundwater sample will be collected from inside the drive rod using a disposable bailer.
- The sampling device will be removed from the borehole and decontaminated prior to reuse at the next depth interval.

Temporary Mini-Well Technique – The following steps will be followed to collect grab groundwater samples using the temporary mini-well technique:

- The drilling subcontractor will advance the borehole to the desired grab groundwater sampling interval.
- If necessary, drill rods will be removed from the borehole.
- Temporary steel or PVC well casing will be lowered into the well. The lowermost 5 foot section will consist of factory slotted casing.
- The drill casing will be used as tremie pipe and pulled back as a gravel pack is emplaced

to at least 2 feet above the screen interval. After confirming the height of the gravel pack using a weighted tag line, a bentonite seal will be emplaced on top of the gravel pack and allowed to hydrate.

- Water level will be recorded in the temporary mini-well using an electronic sounder.
- The mini-well will be developed by bailing or pumping and the water level recorded at the completion of development.
- A grab groundwater sample will be collected using a disposable bailer.

3.4.1 SAMPLE COLLECTION

The sampling hydrogeologist will wear nitrile gloves while collecting samples. The water from the disposable bailer will be transferred into three to VOA vials by holding the VOA vial at an approximate 45 degree angle to minimize turbulence will pouring the water into the vial. The container will be filled to slightly overflowing, forming a convex meniscus at the mouth of the container. The cap will be placed upon the convex meniscus and screwing on tightly to seal the container. To check that the sample is air free, the container will be inverted gently and the cap gently tapped. The absence of entrapped air indicates a successful seal. When air is encountered in the container, a small amount of water from the bailer will be added to the VOA vial and the container will be rechecked for entrapped air.

3.4.2 SAMPLE HANDLING AND CUSTODY

The groundwater samples will be handled using the sample handling procedures described in Section 2.3.3 of this SAP.

3.4.3 SAMPLE DOCUMENTATION

A Groundwater Sampling form will be filled out for all samples taken. The form will include the following information:

- Boring Identification.
- Depth of sample collection
- Method of sample collection.

- Type of samples taken.

A COC form will be completed after all groundwater samples are collected. The COC will accompany the groundwater samples through the sampling day and will be delivered to the laboratory with the samples. Upon delivery to the laboratory, the COC will be relinquished to the laboratory representative accepting the groundwater

3.5 EQUIPMENT DECONTAMINATION PROCEDURES

Large sized drilling and groundwater sampling equipment (e.g. the drill rig and casing), will be decontaminated by washing with a high-pressure hot water spray (aka “steam cleaning”). Steam cleaning will be performed in the designated area on the former Romic facility.

Smaller portable equipment may be decontaminated by hand prior to re-use using a mild solution of detergent (such as Alconox) followed by a double rinse.

The methods used for equipment decontamination will be documented in the field notes.

3.6 VAPOR RISK SCREENING BORING DRILLING

One soil boring will be drilled and sampled at a representative location at the former Romic facility to assess the level of risk associated with the soil vapor for current and future indoor exposure scenarios. The borehole will be advanced to an approximate depth of 40 feet bgs. The total drill depth is subject to change based on site conditions observed by the hydrogeologist. Borehole cuttings will be collected at a minimum of 5-foot intervals for lithologic examination and soil sample collection for geotechnical testing using ASTM procedures. Samples will be collected for analysis for soil bulk density (ASTM D2937), grain density (ASTM D854), soil moisture content (ASTM D2216) and particle size analysis (ASTM D422).

Equipment used during sample collection will include a split-spoon drive assembly and brass ring liners. New brass liners that have decontaminated in accordance with the procedures set forth in Section 4.6 will be used for the collection of the soil samples submitted for laboratory analyses. Alternate sampling equipment may be used depending on drill rig capabilities, but must be authorized by the on-site hydrogeologist. The split-spoon sampler is lowered by the drill rigs cable works inside the drill casing and driven into the native soils below the drill casing.

The brass ring liners are removed and capped from the sampler for geotechnical testing.

3.7 HANDLING OF INVESTIGATION DERIVED WASTE

The drill cuttings and fluids generated during the borehole advancement will be contained at the surface and placed in roll-off type bins located at the former Romic facility or an alternate location provided by GRIC. A composite sample of the drill cuttings and a sample the drilling fluids will be collected from each container and submitted for analysis. Composite samples will be collected by visually dividing each roll-off bin into four quadrants and collecting a portion of sample from each quadrant with a hand auger or similar device. Samples will be preserved by sealing the ends of the brass liner and placing on ice and transported to the laboratory for compositing and analysis. Samples will be analyzed for VOCs by EPA Method 8260, SVOCs by EPA Method 8270, and the RCRA metals by EPA Methods 6020 and 7471A (mercury). Disposal will be arranged based on the analytical results. Steam cleaning will be performed at the designated pad on the Romic facility and the fluids contained and disposed of by Romic. The water and detergent solution used for cleaning smaller pieces of equipment will be disposed of in the sanitary sewer.

4.0 MONITOR WELL CONSTRUCTION

If the results of the grab groundwater sampling are interpreted as justification for installing a new monitoring well following accelerated consultation with EPA and GRIC-DEQ, then the well will be installed in accordance with the general procedures described in this section. Monitoring well installation is subject to access approval from the appropriate GRIC entities. If the vertical profile sampling from any of the locations indicates that the bottom of the plume extends deeper than 120 feet bgs, then a new deeper monitoring well will be installed adjacent to existing well LB-10. If it is determined, based on the vertical profile results from the supplemental boring, that the base of the plume extends below the existing monitor well network depth, then the supplemental boring location will be converted into a permanent monitoring well. After completion, the newly constructed monitor well will be developed to remove fine sediments from the well and annular material. The construction and development of the monitor well will be overseen by the on-site hydrogeologist.

4.1 WELL CONSTRUCTION PROCEDURES

Monitor wells will be constructed in accordance with the requirements of the GRIC-DEQ Well Construction Decommissioning Regulations, version 1.7 (GRDEQ, 2007). The top twenty feet of the wells will be constructed of 4-inch flush threaded Schedule 40 PVC well screen with 0.020-inch factory cut slots. The exact length and depth of the well screen will be based on the results of the grab groundwater sampling described above. The well screen will consist of 4-inch flush threaded Schedule 40 PVC well screen with 0.020-inch factory cut slots. A filter pack of 10-20 sieve size silica sand will be installed across the screen zone and extend approximately 10 feet above the screen zone to prevent the penetration of the seal material into the well. A 10 foot bentonite seal will be placed above the filter pack using a bentonite seal and hydrated with potable water. A slurry of bentonite grout will be emplaced above the bentonite seal up to 20 feet bgs and a cement seal will be placed from 20 feet bgs to ground surface. The well will be completed at the surface in a flush-mounted traffic-rated well vault and secured with a locking waterproof seal at the well head. The well will be surveyed to provide location and elevation information to the nearest 0.01 foot consistent with GRIC datum. The proposed well construction diagram is shown on Figure 3.

4.2 WELL DEVELOPMENT PROCEDURES

Following well completion, the well will be developed using a combination of the swab and bail method and pumping. The well will first be swabbed with a swab block containing a rubber flange nearly the size of the well's inner diameter. The swabbing action removes fine-grained sediment from the filter pack material and assists in settling and removing void spaces from the filter pack. Sediment will then be removed from the well using a bailer. Well development will be conducted until physical water parameters (temperature, pH, and conductivity) stabilize to within $\pm 5\%$ and turbidity decreases as near as practical to 5 Nephelometric Turbidity Units (NTUs). Swab and bail development is anticipated to last one to two hours. After swabbing and bailing is complete, the well will be pumped until the water is clear and free of any visible sediment. The on-site hydrogeologist may instruct the driller to use an alternate method, such as airlifting, if site conditions make an alternate method necessary. All water generated by the well installation and development will be contained onsite either at the former Romic facility or an alternate location provided by GRIC until characterized for disposal.

4.3 WELL CONSTRUCTION DOCUMENTATION

The hydrogeologist and driller will both maintain a schematic of the well specifications as designed by a registered geologist. The hydrogeologist will maintain a pipe tally documenting the well casing materials installed and a record of the annular materials installed during well construction. The hydrogeologist will maintain a field log to document any other relevant information related to well construction.

5.0 REPORTING

Upon completion of the investigation activities, a RFI report will be compiled and will include the following:

- Chronology of drilling and groundwater sampling activities;
- Photographs of drilling and groundwater sampling activities;
- Lithologic log containing geologic descriptions of drill cutting samples using the Unified Soil Classification System;
- Field notes compiled by the on-site hydrogeologist during drilling operations;
- Analytical laboratory results for the water samples collected from the site;
- A comparison of detected concentrations of constituents to EPA's PRGs and MCLs with an identification of results exceeding standards or guidance levels; and

If applicable,

- Chronology of drilling, monitoring well installation and development activities;
- Photographs of drilling, monitoring well installation and development activities;
- Schematic as-built well construction diagram illustrating well construction details;
- Well survey information;

In addition, the report will include a summary screening of the preliminary corrective measures described in the RFI Work Plan. Integrated into the RFI data analysis will be an update to the CSM and a reassessment of the vertical and horizontal extent of the northern plume. A possible outcome of the assessment may be the determination of whether installation of a distal compliance monitor well is necessary for regulatory purposes. Romic will provide EPA the RFI Report approximately 30-days after receiving the final laboratory reports from the groundwater sampling activities.

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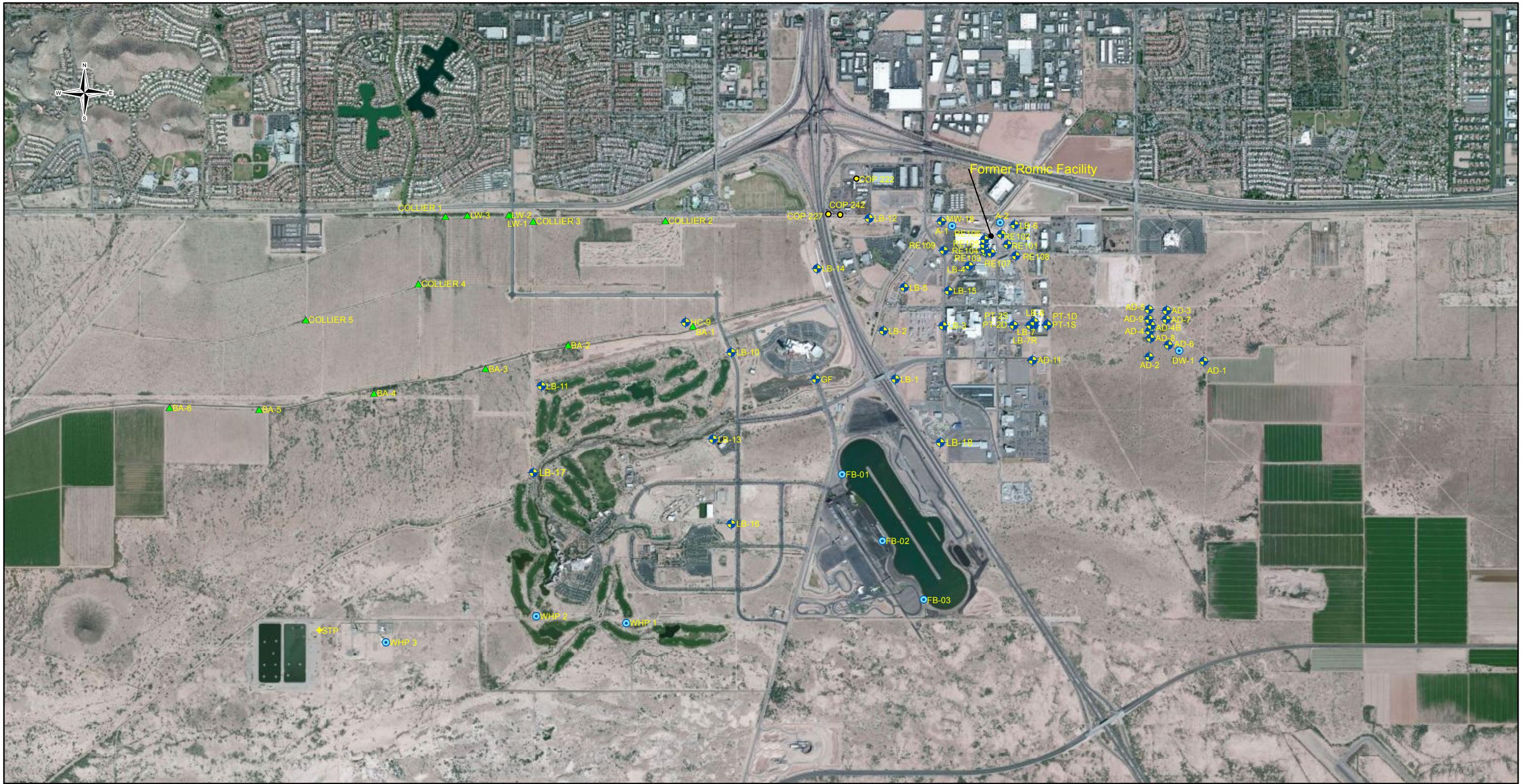
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LFR, Inc., 2009. Deep Soil Gas and Groundwater Sampling Summary Report, Romic Environmental Technologies Corporation, March 18, 2009.

FIGURES



VICINITY / WELL LOCATION MAP

Explanation

- Monitor Well
- ▲ Agricultural Well
- Drinking Water Well
- COP
- POC Well

0 0.25 0.5 1 1.5 Miles

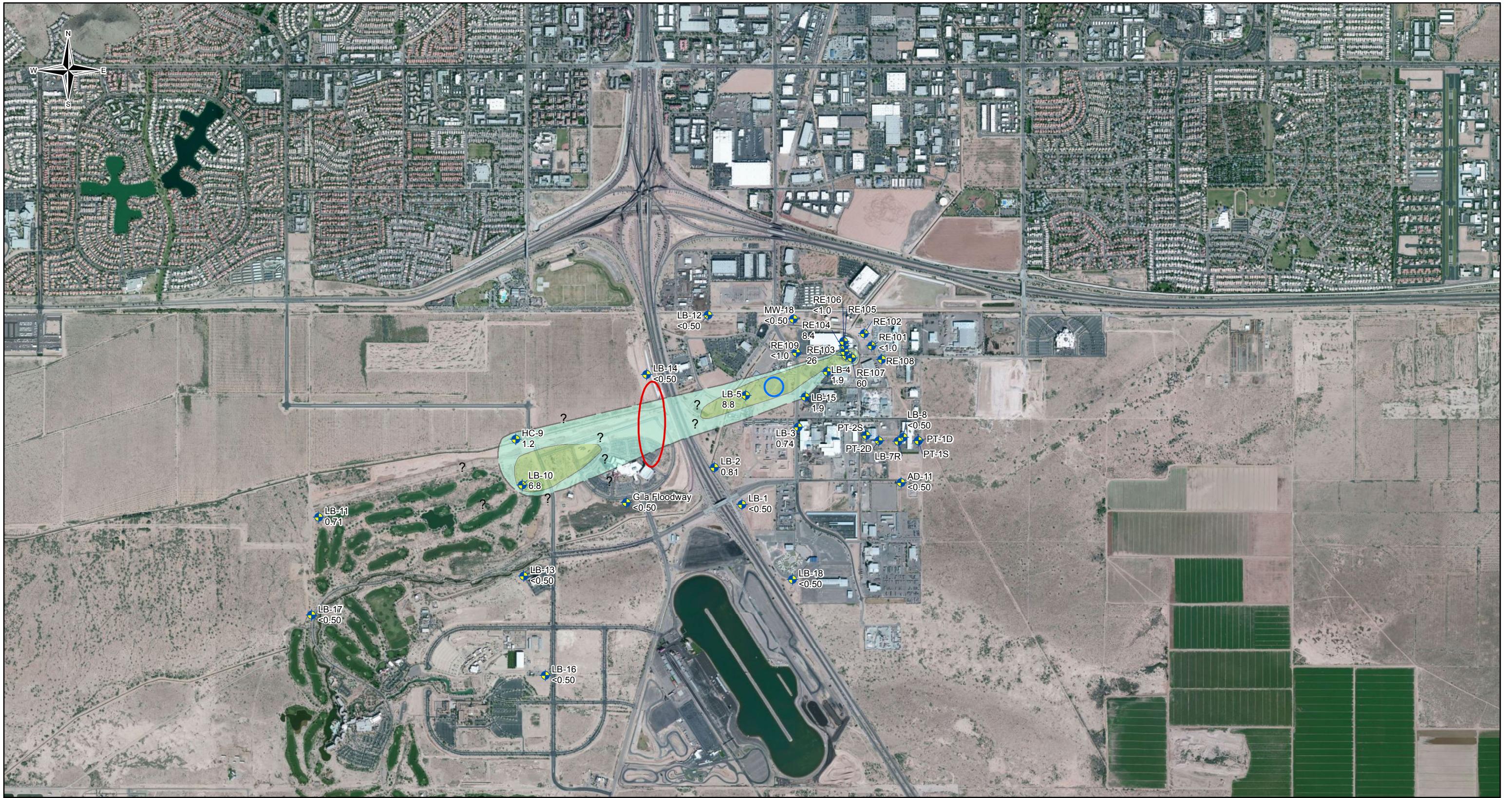
6155 East Indian School Road
Suite 200
Scottsdale, Arizona 85251
(480) 659-7131

CLEAR CREEK ASSOCIATES

RCRA Facility Investigation (RFI) Sampling and
Analysis Plan (SAP)
Former Romic Environmental Technologies Facility

Figure 1

December 2011



Explanation

◆ August 2011 PCE Concentration (ug/L)

PCE Concentration Contours

- >1.0 ug/L
- >5.0 ug/L
- >25 ug/L
- >50 ug/L

World Imagery

○ Proposed Transect Location

○ Proposed Boring Location

Areas of Additional Investigation

0 0.25 0.5 Miles

6155 East Indian School Road
Suite 200
Scottsdale, Arizona 85251
(480) 659-7131

CLEAR CREEK ASSOCIATES

RCRA Facility Investigation (RFI) Sampling and Analysis Plan
Former Romic Environmental Technologies Facility

Figure 2

December 2011

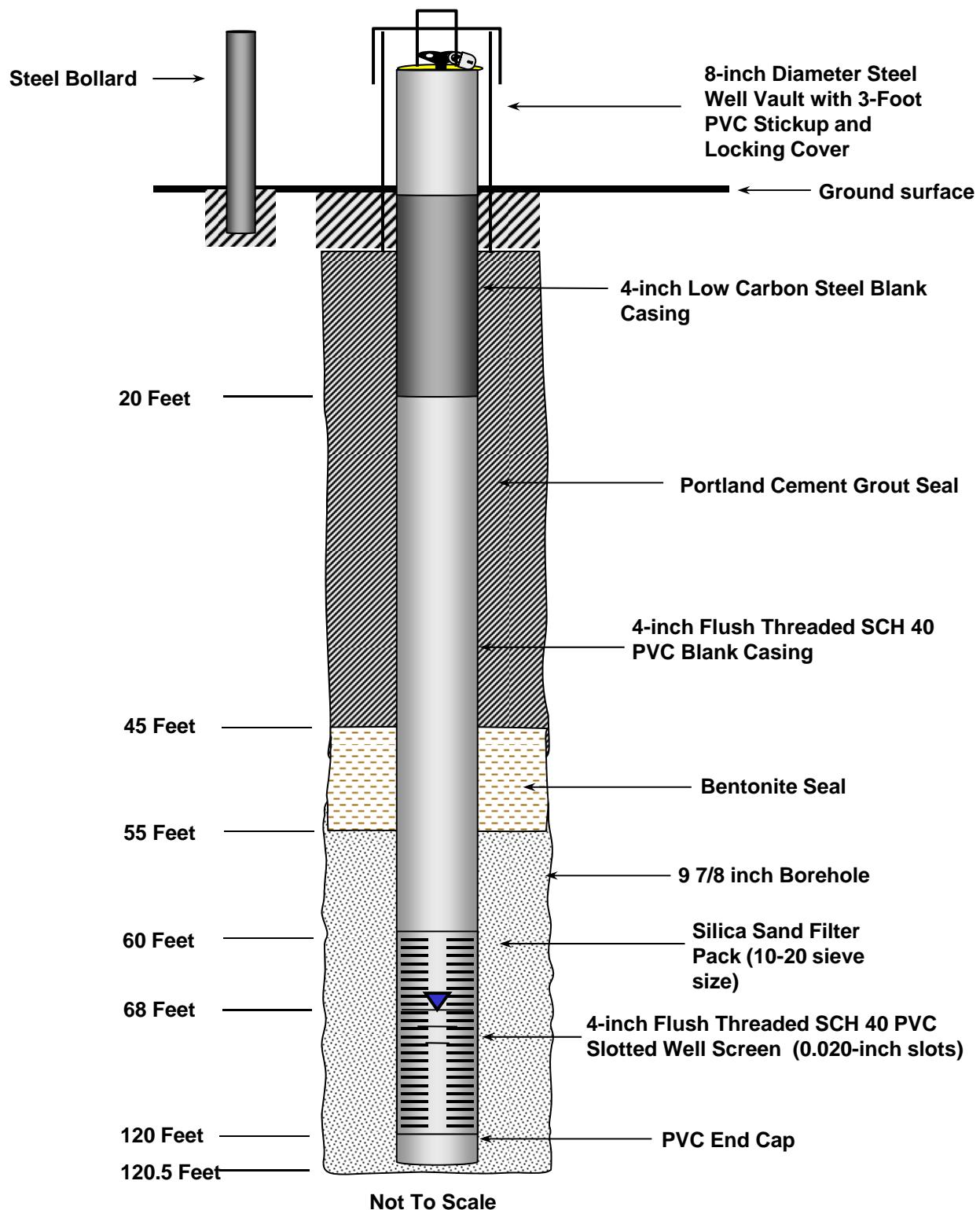


FIGURE 3
PROPOSED MONITOR WELL DESIGN
 RCRA Facility Investigation
 Sampling and Analysis Plan
 Romic Environmental Technologies
 Gila River Indian Community, Arizona

TABLES

TABLE 1
VOLATILE ORGANIC COMPOUNDS
RCRA Facility Investigation (RFI) SAP
 Romic Environmental Technologies
 Gila River Indian Community, Arizona

Analyte	Analytical Method
Acetone	EPA 8260B
Acetonitrile	EPA 8260B
Acrolein (Propenal)	EPA 8260B
Acrylonitrile	EPA 8260B
Allyl alcohol	EPA 8260B
Allyl chloride	EPA 8260B
Benzene	EPA 8260B
Benzyl chloride	EPA 8260B
Bis(2-chlorethyl)sulfide	EPA 8260B
Bromoacetone	EPA 8260B
Bromochloromethane	EPA 8260B
Bromodichloromethane	EPA 8260B
4-Bromofluorobenzene	EPA 8260B
Bromoform	EPA 8260B
Bromomethane	EPA 8260B
n-Butanol	EPA 8260B
2-Butanone	EPA 8260B
t-Butyl alcohol	EPA 8260B
Bromobenzene	EPA 8260B
Butylbenzene	EPA 8260B
Carbon disulfide	EPA 8260B
Carbon tetrachloride	EPA 8260B
Chloral hydrate	EPA 8260B
Chloroacetonitrile	EPA 8260B
Chlorobenzene	EPA 8260B
1-Chlorobutane	EPA 8260B
Chlorodibromomethane	EPA 8260B
Chloroethane	EPA 8260B
2-Chloroethanol	EPA 8260B
2-Chloroethyl vinyl ether	EPA 8260B
Chloroform	EPA 8260B
1-Chlorohexane	EPA 8260B
Chloromethane	EPA 8260B
Chloroprene	EPA 8260B
3-Chloropropionitrile	EPA 8260B
2-Chlorotoluene	EPA 8260B
4-Chlorotoluene	EPA 8260B
Crotonaldehyde	EPA 8260B
1,2-Dibromo-3-chloropropane	EPA 8260B
1,2-Dibromoethane	EPA 8260B



TABLE 1 (cont.)
VOLATILE ORGANIC COMPOUNDS
Groundwater Monitor Well Installation SAP
Romic Environmental Technologies
Gila River Indian Community, Arizona

Analyte	Analytical Method
Dibromofluoromethane	EPA 8260B
Dibromomethane	EPA 8260B
1,2-Dichlorobenzene	EPA 8260B
1,3-Dichlorobenzene	EPA 8260B
1,4-Dichlorobenzene	EPA 8260B
cis-1,4-Dichloro-2-butene	EPA 8260B
trans-1,4-Dichloro-2-butene	EPA 8260B
Dichlorodifluoromethane	EPA 8260B
1,1-Dichloroethane	EPA 8260B
1,2-Dichloroethane	EPA 8260B
1,1-Dichloroethene	EPA 8260B
cis-1,2-Dichloroethene	EPA 8260B
trans-1,2-Dichloroethene	EPA 8260B
1,3-Dichloropropane	EPA 8260B
2,2-Dichloropropane	EPA 8260B
1,1-Dichloropropene	EPA 8260B
1,2-Dichloropropane	EPA 8260B
1,3-Dichloro-2-propanol	EPA 8260B
cis-1,3-Dichloropropene	EPA 8260B
trans-1,3-Dichloropropene	EPA 8260B
1,2,3,4-Diepoxybutane	EPA 8260B
Diethyl ether	EPA 8260B
1,4-Difluorobenzene	EPA 8260B
1,4-Dioxane	EPA 8260B
Epichlorhydrin	EPA 8260B
Ethanol	EPA 8260B
Ethyl acetate	EPA 8260B
Ethylbenzene	EPA 8260B
Ethylene oxide	EPA 8260B
Ethyl methacrylate	EPA 8260B
Fluorobenzene	EPA 8260B
Hexachlorobutadiene	EPA 8260B
Hexachloroethane	EPA 8260B
2-Hexanone	EPA 8260B
2-Hydroxypropionitrile	EPA 8260B
Iodomethane	EPA 8260B
Isobutyl alcohol	EPA 8260B
Isopropylbenzene	EPA 8260B
Isopropyltoluene	EPA 8260B
Malononitrile	EPA 8260B



TABLE 1 (cont.)
VOLATILE ORGANIC COMPOUNDS
Groundwater Monitor Well Installation SAP
Romic Environmental Technologies
Gila River Indian Community, Arizona

Analyte	Analytical Method
Methyl acrylate	EPA 8260B
Methacrylonitrile	EPA 8260B
Methyl-t-butyl ether	EPA 8260B
Methanol	EPA 8260B
Methylene chloride	EPA 8260B
Methyl methacrylate	EPA 8260B
4-Methyl-2-pentanone	EPA 8260B
Naphthalene	EPA 8260B
Nitrobenzene	EPA 8260B
2-Nitropropane	EPA 8260B
N-Nitoso-di-n-butylamine	EPA 8260B
Paraldehyde	EPA 8260B
Pentachloroethane	EPA 8260B
Pentafluorobenzene	EPA 8260B
2-Pentanone	EPA 8260B
2-Picoline	EPA 8260B
1-Propanol	EPA 8260B
2-Propanol	EPA 8260B
Propargyl alcohol	EPA 8260B
B-Propiolactone	EPA 8260B
Propionitrile (Ethyl cyanide)	EPA 8260B
n-Propylamine	EPA 8260B
n-Propylbenzene	EPA 8260B
Pyridine	EPA 8260B
Styrene	EPA 8260B
1,1,1,2-Tetrachloroethane	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 8260B
Tetrachloroethene	EPA 8260B
1,2,3-Trichlorobenzene	EPA 8260B
Trichloroethylene	EPA 8260B
Trichlorofluoromethane	EPA 8260B
1,2,3-Trichloropropane	EPA 8260B
1,2,4-Trimethylbenzene	EPA 8260B
1,3,5-Trimethylbenzene	EPA 8260B
Vinyl acetate	EPA 8260B
Vinyl chloride	EPA 8260B
Xylene	EPA 8260B

ATTACHMENT A

FIELD DATA FORMS

DAILY FIELD REPORT

Job Number:	
Page	of
Date:	Day
Staff:	HRS Charged



DAILY FIELD REPORT

Job Number:

Date: _____ **Day:** _____

Page _____ **of** _____



Project No. _____

Well/Boring _____
Page ____ of ____

Project/Client Name	Location (Cadastral)		Elevation (ft amsl)	Project No.
Drilling Co.	Location (NAD 83 GPS Latitude Longitude)		Date Started	Date Finished
Lithology Described By	Drilling Equipment	Drilling Method	ADWR Well Registration No.	
Total Depth	Drilling Fluid		* Indicates (based on visual estimates of volume):  Relative % fines (F < 0.074mm)  Relative % sand (S>.074<4.8mm)  Relative % gravel (G > 4.8mm)	
Bit Diameter	Conductor Casing (type; diameter; depth)			
Comments				
* Classification System:	Unified Soil Classification System (ASTM)			
Description	Depth (feet)	Drill Rate (feet/hour)	* F S G (%)	Remarks

Project No. _____

Well/Boring _____
Page ____ of ____

Description	Depth (feet)	Drill Rate (feet/hour)	* F S G (%)	Remarks
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			
-	-			



PIPE TALLY

Project Name.:	Project No.:
Well No.:	Date:
Location:	Pipe Tally for:
Total Depth:	Geologist:

Type of Connections: Welded T+C Flush Thread Other

Pipe	✓	Length (ft)	Length Σ (ft)	Pipe Type	Pipe	✓	Length (ft)	Length Σ (ft)	Pipe Type
SUMMARY OF TALLY									
Total length of casing/screen tallied (ft.): _____									
Length of casing cut off after landing (ft.): _____									
Bottom of Casing (feet below land surface): _____									
Screened Interval(s) (ft.bls): _____									
Total feet of blank casing in hole (ft.): _____									

Notes:

ESTIMATED ANNULAR MATERIAL RECORD

GROUNDWATER SAMPLING FORM

LOCATION		Well Number (Site ID):
Project Name:		Date:
Project Number:		Samplers:
Location:		Weather:

INSTRUMENTATION		Instruments calibratated on:	Date:	Time:
pH Meter:				
pH calibration readings at:		4.0:	7.0:	10.0:
Conductivity Meter:		Calibration solution used:	Reading:	

WELL INFORMATION	Depth to water (DTW):	feet bmp	Date:	Time:
Total Depth of Well (TD):	feet bmp	Screened Interval:	Casing Diameter:	
Casing Type:				

$$(\underline{\quad} - \underline{\quad}) \times \underline{\quad}^2 \times \underline{\quad} \times 0.0408 = \underline{\quad}$$

*TD DTW Well Dia. Casing
 Volumes*

WELL EVACUATION			Purge Equipment:								
CLOCK TIME	ELAPSED TIME (min)	PUMPING RATE (gpm)	PURGE VOLUME (gal)	DTW (feet)	pH	Specific Cond. (mS/cm)	Temp C° F°	COLOR	CLARITY	ODOR	

SAMPLE COLLECTION			Date:	Time:	Volume Purged:	gal.			
Requested Analysis:			Collection Equipment:						
Containers:									
Filtered?:	<input type="checkbox"/>	Yes <input type="checkbox"/>	No	Filter Equipment:	SAMPLE TYPE:				
Comments:						<input type="checkbox"/>	Primary	<input type="checkbox"/>	Duplicate
						<input type="checkbox"/>	Equipment Blank	<input type="checkbox"/>	Field Blank
						<input type="checkbox"/>	Split Sample	<input type="checkbox"/>	MS/MSD
Sampler Signature:			Date:						



L - A - B S - C - I - E - N - C - E - S
12065 Lebanon Road
Mt. Juliet, TN 37122

Phone: (800) 767-5859
Phone: (615) 758-5858
Fax: (615) 758-5859

Analysis/Container/Preservative

Billing Information:

Report to:
Email to:

Project Description:
City/Site Collected

Client Project #:

ESC Key:

Collected by:
Site/Facility ID#:

P.O.#:

(lab use only)

Template/Prelogin
Shipped Via:

(lab use only)

CoCode
Remarks/Contaminant

(lab use only)

Sample # (lab only)

Rush? (Lab MUST Be Notified)
 Same Day 200%
 Next Day 100%
 Two Day 50%

Date Results Needed:

No.
 Email? No Yes
 FAX? No Yes

of

Cntrs

Comp/Grab

Matrix*

Depth

Date

Time

Sample ID

Received by: (Signature)

Date:

Time:

Condition:

Received by: (Signature)

Date:

Time:

*Matrix: **SS** - Soil/Solid **GW** - Groundwater **WW** - WasteWater **DW** - Drinking Water **OT** - Other _____

Remarks:

pH _____

Flow _____

Temp _____

Other _____

(lab use only)

Condition: _____

UPS
 FedEx Courier

Coc Seals Intact: Y N NA

Sample # (lab only)

Relinquished by: (Signature)

Received by: (Signature)

Date:

Date:

Time:

Time:

Temp:

Temp:

Bottles Received:

Time:

pH Checked:

NCF:

SPEEDIE & ASSOCIATES LABORATORY WORK REQUEST

PROJECT: _____ PROJECT NO: _____

PROJECT LOCATION: _____ LAB SAMPLE NO: _____

CLIENT: _____ SAMPLED BY: _____ SUBMITTED BY: _____
 Native Import

MATERIAL SOURCE: _____ DATE SAMPLED: _____ DATE SUBMITTED: _____
 ABC Sand Aggregate Asphalt Soil Other

MATERIAL DESCRIPTION: _____

SAMPLE LOCATION: _____

TESTS REQUIRED/AGENCY: _____ DUE DATE: _____

REQUESTED BY: _____ SAMPLE QUANTITY/CONTAINER: _____

ATTACHMENT B

PID CALIBRATION PROCEDURES

Scorpion Vapor Meter

Model AX-1 PID

Standard Operating Procedure

Basic Operation

Introduction

The Scorpion Vapor Meter AX-1 PID is used for the detection and concentration monitoring of photo-ionizable vapors in air. Regardless of the type of monitoring task for which the Scorpion will be used, a standard set of operating procedures should be followed in preparation to perform these tasks. This document outlines and explains the steps necessary to set up the Scorpion to perform its basic function, to detect and measure the concentration of photo-ionizable vapors in air. It is recommended that the Scorpion Vapor Meter AX-1 Instrument Manual be reviewed to familiarize the operator with the details of the functions and features and physical layout of the instrument.

Instrument Inspection and Battery Charging

Before the Scorpion can be used a brief inspection is required as listed below:

- 1- Remove the inlet filter cover to verify the element is present. If it is missing or if it is contaminated install a new element.
- 2- Charge the battery by plugging one of the Scorpion Power sources into the Power connector at the rear of the instrument housing. A completely discharged battery will take 2.5 hours to charge. A fully charged battery will provide 25 hours of continuous monitoring.
- 3- Make sure all covers are securely fastened: Inlet filter housing, Detector lamp cover, Sample Conditioning Port cover, Battery, Communication and Power connector dust covers

Instrument Power Up

The following outline explains how to power the Scorpion On to take vapor concentration readings:

- 1- The Scorpion is Powered On by depressing any key in the 'ON' key column. A 'self-test' will run for several seconds.
- 2- After the self-test is completed the Scorpion will be in the Monitor Mode ready to make vapor concentration readings. Depress the 'ON' key now to ignite the PID lamp and start the vacuum pump. Concentration readings can now be made.
- 3- To turn the Scorpion Off Depress the 'ON' key while in the Monitor Mode. The PID lamp and vacuum pump will turn off. In a few minutes the Scorpion will turn off automatically.

NOTE: PID calibration will have to be performed if it has not yet been done or if the instrument has been used for many hours of monitoring.

Calibration

In order to make accurate vapor concentration readings the Scorpion must be calibrated periodically, or at least have its calibration checked with a calibration gas standard. The following steps outline how to calibrate the Scorpion:

- 1- Turn the Scorpion on. Several seconds after the self-test the Scorpion will be in the Monitor Mode.
- 2- Depress the 'ESC' key and the Main Menu will be displayed.
- 3- Depress the '6' key to select the CALIBRTN item. Once selected it will be highlighted in UPPER CASE.
- 4- Depress the 'ENT' key to enter and display the calibration mode.
- 5- Depress the '1' key to select the CREATE option and depress 'ENT'.
- 6- The display will now prompt for entry of the concentration of the calibration gas to be used, referred to as SPAN GAS. Using the numeric keys enter the concentration and then depress 'ENT'.
- 7- The display will now prompt for the calibration gas to be connected to the Scorpion's inlet. If using a cylinder / regulator standard: attach the calibration fitting to the inlet and inset the regulator into it. Turn the gas on then depress 'ENT'. The Scorpion will indicate that it is calibrating. Alternate cal-gas connections are covered in the Instrument Manual.
- 8- Upon completion of the Span calibration the display will ask if additional Span points are required. If additional span points are to be used depress "ENT" and go to step "6" above. Otherwise depress 'CLR'. Remove and turn off the SPAN GAS.
- 9- The display will now prompt for the connection of ZERO GAS. Introduce a source of ZERO GAS, either a cylinder or a location free of vapors, to establish the zero calibration point. Depress 'ENT'.
- 10- The display will indicate that it is calibrating the ZERO point. Upon completion the display will prompt for the entry of the lamp energy and lamp I.D.. Enter these values using the numeric keys and depress 'ENT'. This is optional information used for labeling the calibration data.

Additional Operating Procedures

The Scorpion Vapor meter is capable of many sophisticated monitoring and data logging tasks. Usage, setup procedures, and examples are provided in the Instrument Manual and in additional SOPs particular to each monitoring task.

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN



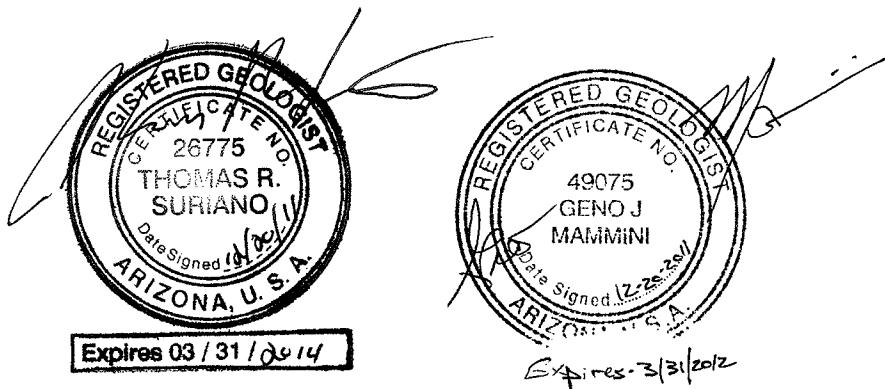
RCRA Facility Investigation (RFI) Work Plan
Former Romic Environmental Technologies Facility
Gila River Indian Community, Arizona

December 2011
212001

RFI WORK PLAN APPENDIX B

QUALITY ASSURANCE PROJECT PLAN RCRA FACILITY INVESTIGATION AND CONTINUED GROUNDWATER SAMPLING

FORMER ROMIC ENVIRONMENTAL TECHNOLOGIES FACILITY
LONE BUTTE INDUSTRIAL PARK
GILA RIVER INDIAN COMMUNITY, ARIZONA



Prepared By

Clear Creek Associates
6155 E. Indian School Rd., Suite 200
Scottsdale, Arizona 85251

December 2011
Project No. 212001

In Participation with

Iris Environmental
1438 Webster Street, Suite 302
Oakland, California 94612



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LIST OF ATTACHMENTS

Attachment A Environmental Science Corp. Standard Operating Procedures

Attachment B Environmental Science Corp. List of Analytes and Reporting Limits for EPA Method 8260B

Attachment C Arizona Department of Health Services Arizona Data Qualifiers Guidance Document

Attachment D Chain of Custody and Laboratory Work Request Forms



1.0 INTRODUCTION

Clear Creek Associates PLC (Clear Creek) and Iris Environmental (Iris) on behalf of Romic Environmental (Romic) have prepared this Quality Assurance Project Plan (QAPP) for the former Romic Environmental facility, located at Lone Butte Industrial park, Gila River Indian Community, Arizona (Site). This QAPP establishes the methods for management of the field program described in the Sampling and Analysis Plan (SAP) included as Appendix A to the *RCRA Facility Investigation (RFI) Work Plan* (Work Plan) so that collection of valid and appropriate data will occur and will meet the project objectives.

The purpose of the QAPP is to specify the quality assurance (QA) organization and functional activities associated with site characterization work as well as the quality control (QC) criteria that will allow the data quality objectives (DQOs) to be achieved. The primary focus of the work covered in this QAPP is to perform a validated, screening level assessment of volatile organic compounds (VOCs) in groundwater at the site and surrounding area. An additional goal of this work is to perform a risk-based evaluation of geotechnical characteristics of site soils in one representative location.

Field activities will be performed by Iris and Clear Creek and their subcontractors in accordance with the procedures set forth in the SAP and this QAPP. Environmental Science Corporation (ESC) has been selected as the analytical laboratory for VOC analyses and Speedie and Associates has been selected as the geotechnical laboratory. Subcontractors for the drilling work have not been determined.

1.1 QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVES

The QA program is designed to ensure that the precision, accuracy, completeness, comparability, and representativeness of data generated during the project meet the defined DQOs, see Section 3 of this QAPP. QC is the routine application of procedures for achieving prescribed performance standards (in monitoring and measurement) during project planning, field activities, sample analysis, sample and data handling, and data evaluation and interpretation. Together, QA/QC ensure that the work conducted during a project will fulfill the project's DQOs.

The sampling program for this site is formally described in the Work Plan. This QAPP contains general and specific details regarding field sampling, laboratory, and analytical procedures that apply to activities described in the Work Plan. It provides field and laboratory personnel with instructions regarding activities to be performed before, during and after field investigations. These instructions will ensure data collected for use in project decisions will be of the type and quality needed for their intended purpose.

1.2 QAPP OBJECTIVES AND USE

Standard procedures and specifications are established to ensure that the laboratories produce comparable data, and that data quality is consistently assessed and documented. The specific objectives of this QAPP are to:

- Provide standardized references and quality specifications for all anticipated field sampling, analysis, and data review procedures required for the Site;
- Provide guidance and criteria for selected field and analytical procedures; and
- Establish procedures for reviewing and documenting compliance with field and analytical procedures.

1.3 SOURCES OF INFORMATION

This QAPP has been prepared using information from the following documents:

- United States Environmental Protection Agency (EPA QA/G-4), *Guidance for the Data Quality Objectives Process*, February 2006 (USEPA 2006)
- United States Environmental Protection Agency (EPA QA/R-5), *EPA Requirements for Quality Assurance Project Plans*, March 2001 (USEPA 2001)

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

This section identifies the persons and organizations involved in the project and presents their specific roles and responsibilities. Project management responsibilities include QA/QC process oversight and implementation. The following sections identify the key participants at the U.S. Environmental Protection Agency (EPA), the Gila River Indian Community Department of Environmental Quality (GRIC-DEQ), the Lone Butte Industrial Development Corporation (LBIDC), Romic, Iris, Clear Creek, and subcontractors and include descriptions of each participant's QA/QC responsibilities.

EPA and GRIC-DEQ

The EPA Project Manager, Mr. John Moody, has EPA oversight responsibility for corrective action work conducted at the Site.

Mr. Glen Stark has GRIC-DEQ oversight responsibility for work conducted at the Site.

Romic Environmental

Mr. Christopher Alger (Iris) is the designated Project Coordinator for corrective action on behalf of Romic, in accordance with the Administrative Order upon Consent. Mr. Alger will coordinate with Romic management as necessary to ensure that the necessary resources are available and that project objectives and standards are addressed.

Iris Environmental and Clear Creek

Iris is contracted to Romic to oversee and participate in corrective action activities. Mr. Christopher Alger is the Iris Project Coordinator and is responsible for ensuring Iris personnel adhere to the procedures set forth in the SAP and QAPP.

Clear Creek is the field program implementation lead and will provide field geologist to oversee the field program. Mr. Tom Suriano is the Clear Creek Project Manager. Mr. Suriano is responsible for ensuring that Clear Creek personnel adhere to the procedures set forth in the SAP and QAPP.

Mr. Geno Mammini of Clear Creek is the Field Team Supervisor and the Project QA Coordinator. Mr. Mammini will be responsible for laboratory coordination, sampling, and sample documentation. Mr. Mammini reports to the Clear Creek project manager.

Mr. Dave Giles is Clear Creek's Project Health & Safety (H&S) officer. Mr. Giles is responsible for developing, implementing and updating the Health and Safety Plan (HSP). Mr. Giles reports to the Clear Creek project manager.

Mr. Alger and Mr. Suriano will jointly coordinate the field team.

Quality Assurance Coordinator and Laboratory Responsibilities

The Project QA Coordinator and laboratory personnel have shared responsibilities to coordinate analytical activities and maintain control of samples at all times. QA procedures and established protocols must be followed to meet project objectives.

Clear Creek Project QA Coordinator

The Project QA Coordinator is responsible for applying the project QA program as it relates to the collection and completeness of field and laboratory data, field personnel oversight, and compliance with established data quality control practices. The Project QA Coordinator coordinates delivery of groundwater and soil samples to the analytical and geotechnical laboratories, manages receipt of data from the laboratories, performs data review tasks, communicates with the Project Manager regarding field and laboratory data review concerns, and reviews reports and documentation generated from the program.

Analytical Laboratory

Environmental Science Corp. (ESC) of Mount Juliet, Tennessee has been selected to perform chemical analyses of groundwater samples as described in the Work Plan. ESC has a Quality Assurance Manual that has been reviewed by the Project Manager and the Project QA Coordinator and found to be adequate to achieve the quality objectives for this project. The ESC QA Manager will oversee analytical activities and will verify that laboratory protocols are being followed in accordance with their QA Manual, this QAPP, and the Work Plan. The ESC QA Manager will review each chemical data package prior to submission to Clear Creek, and will be responsible for reporting significant and relevant technical deficiencies to the Project QA Coordinator and implementing corrective action. ESC will test groundwater samples for VOCs using EPA Method 8260B. Standard Operating Procedures (SOP) for EPA Method 8260B are included as part of Attachment A.

Geotechnical Laboratory

Speedie & Associates, Inc. of Phoenix, Arizona has been selected to perform geotechnical analyses of soil samples, as described in the Work Plan. Speedie & Associates, Inc. will test soil samples for soil bulk density by American Society for Testing and Materials (ASTM) method D2937, grain density by ASTM D854, soil moisture content by ASTM D2216, and particle size analysis by ASTM D422. Speedie & Associates' Laboratory QA Manager will oversee testing procedures and will verify that laboratory protocols are being followed in accordance with the ASTM standards, this QAPP, and the Work Plan. Any significant and relevant technical difficulties will be reported to the Project QA Coordinator by Speedie & Associates' Laboratory QA Manager who will be responsible for implementing corrective actions.

3.0 SAMPLE ANALYTICAL PROGRAM AND DATA QUALITY OBJECTIVES

The following sections discuss the sample analytical program and DQOs for the RFI. During the RFI sampling program, depth-specific groundwater samples will be collected from existing monitoring wells and from exploratory deep borings at strategic off-site locations. Soil samples will be collected from one on-site geotechnical soil boring. Data obtained from the investigation will be used to address data gaps in the current conceptual site model. As such, DQOs will be developed to facilitate appropriate data collection and validation procedures so that the resulting data will be appropriate for the intended purpose. DQOs are qualitative and quantitative statements that clarify study objectives, define the appropriate type of data, and specify the tolerable levels of potential errors.

3.1 SAMPLE ANALYTICAL PROGRAM

All groundwater samples will be analyzed at ESC for VOCs using EPA Method 8260B as described in the Work Plan. Analytes and reporting limits (RLs) for EPA Method 8260B for groundwater are listed in Attachment B.

All geotechnical samples will be analyzed at Speedie & Associates, Inc. for soil bulk density by ASTM method D2937, grain density by ASTM D854, soil moisture content by ASTM D2216, and particle size analysis by ASTM D422 as described in the Work Plan.

3.2 DATA QUALITY OBJECTIVES

The DQO process is a series of logical steps that guides management or staff to a plan for the resource-effective acquisition of environmental data. The DQO process is used to establish performance and acceptance criteria, which serve as the basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of the study. The DQO process consists of seven iterative steps. The DQOs have been defined for this investigation and are summarized in Table 1. In defining DQOs, various factors were considered including potential uses for the data, the schedule of the project, and overall regulatory requirements. Data generated during this investigation will be used to address data gaps at the Site and to help in the development of a future remedial strategy.

QC criteria are dictated by the project requirements and the intended uses of the data. Chemical data will be used to delineate the northern, distal, and vertical extents of the off-site VOC plume and to quantify soil vapor intrusion risks. When a project objective is defined, as is the case of identifying plume extents, specific analytical methods, reporting limits, and QC criteria can be chosen to minimize bias, imprecision, and decision errors to allow DQOs to be achieved within the specified limits of confidence.

In addition to QC criteria for accuracy and precision, sensitivity was considered when choosing analytical methods to minimize the impact of uncertainty resulting from critical measurements made near the reporting limits. The QA/QC procedures for ESC are described in their Quality Assurance Manual and Standard Operating Procedures for EPA Method 8260B included as Attachment A. Speedie & Associates, Inc. will follow the QA/QC procedures outlined in the ASTM standards. QC data generated by the laboratories will be measured against these criteria. These criteria will continue to be reviewed by the laboratories throughout implementation of the Work Plan, and modified, as necessary and appropriate, in accordance with the guidelines presented in the Quality Control sections of EPA Method 8260B and ASTM Methods D2937, D854, D2216, and D422.

The data will be evaluated by Clear Creek using the criteria presented in Section 6.0 of this QAPP. The comparability of all data will be aided by using consistent data reporting units. Groundwater data will be reported using units of micrograms per liter ($\mu\text{g/l}$). Geotechnical data will be reported in method-appropriate units (e.g. grams per cubic centimeter (g/cm^3) for soil bulk density analysis, etc.).



4.0 SAMPLE COLLECTION AND CUSTODY PROCEDURES

Proposed groundwater and geotechnical soil sampling procedures are specified in the Work Plan. Groundwater samples from existing monitoring wells will be collected using passive diffusion bag (PDB) sampling devices according to the methods outlined in the Sampling and Analysis Plan (SAP). Grab groundwater samples will be collected using a HydroPunch® sampling device or field-constructed temporary well. Geotechnical soil samples will be collected using a lined stainless steel solid barrel or split-spoon sampling device. The QAPP outlines procedures for sample preservation and storage, and data documentation that will be followed to ensure data quality.

4.1 SAMPLE PRESERVATION AND HOLDING TIMES

Groundwater samples will be collected in pre-cleaned, laboratory-supplied, pre-prepared 40-milliliter (ml) glass vials for volatile organic analysis (VOA). All groundwater samples collected for analytical testing will be placed in appropriate containers (either VOA vial holders or resealable bags) and immediately placed on ice in laboratory-supplied coolers and kept cooled until delivered to the laboratory. The hold time for groundwater samples for EPA Method 8260B analysis is 14 days.

Geotechnical soil samples will be collected in a tube lined stainless steel solid barrel or split-spoon sampler. Tubes selected for analyses will be capped at each end and sealed with Teflon® tape. Capped soil samples will be placed in laboratory-supplied coolers and kept cooled and on ice after collection and labeling (until delivered to the laboratory). Samples will be sealed, placed in sealable plastic bags. There are no hold times for soil geotechnical analytical methods.

4.2 SAMPLE LABELING

Sample labels will clearly indicate sample identification, sample date, collection time, sampler's initials, and any pertinent comments such as the type of preservative, if applicable, used on the samples. Sample labels will be completely filled out at the time of sample collection. Sample identifications will be coded.

4.3 SAMPLE DOCUMENTATION AND CUSTODY

After collection, samples will be maintained in the custody of the sampling personnel until formally transferred to another party. For purposes of this work, custody will be defined as follows:

- The sample is in a person's possession.
- The sample is in plain view of the custodian.
- The sample is in a secure area after having been in physical possession.
- The sample is in a designated secure area, restricted to authorized personnel.

Custody seals are not necessary if the samples do not leave the custody of the sampler prior to delivery to the laboratory. If the samples leave the custody of the sampler for any reason, then one or more of each sample container, resealable bag, or each cooler will be sealed with a transportation security seal (chain-of-custody seal) containing the sampler's initials.

COC records, which document sample collection and handling, will be maintained for all samples. Original data recorded in the COC records will be written in waterproof ink. No COC record will be destroyed or discarded, even if it is illegible or contains inaccuracies that require a replacement document. If an error is made on a document compiled by one individual, that individual will make the correction by crossing a line through the error, entering the correct information, and initialing and dating the change. The erroneous information will not be obliterated. Any subsequent error(s) discovered on a document will be corrected by the person discovering the error. All corrections will be initialed and dated.

Example COC and laboratory work request forms are presented in Attachment D for ESC and Speedie & Associates, Inc., respectively. COC forms will be prepared for groups of samples collected on a given day. Each COC form will be prepared in quadruplicate. Two of the four copies will accompany the samples to the laboratory. The yellow copy will be kept in the QA/QC file, and the pink copy will be retained in the project file. The COC form makes provision for documenting sample integrity and the identity of any persons involved in sample transfer. Information entered on the COC will consist of the following:

- project name and number
- COC serial number
- project location
- sample numbers
- sampler/recorder's signature

- date and time of collection of each sample
- collection location
- sample type
- analyses requested
- inclusive dates of possession
- name of person receiving the sample
- laboratory sample number
- date of receipt of sample
- name of laboratory

The COC record will be signed and maintained by the sampling personnel and others who subsequently hold custody of the samples. The COC record will accompany the samples during shipment, if necessary or appropriate.

The laboratory will accept custody of samples and verify that information on the COC form matches the samples received. If any discrepancies are noted, the sample custodian will inform the Project QA Coordinator and discuss any required actions. (Discrepancies may include for example, improper preservation; broken containers; and/or labeling inconsistencies.) The sample custodian will note the physical condition and status of sample preservation upon receipt. The laboratory will maintain a sample receipt form or equivalent to document acceptable condition upon receipt and note any communications made of discrepancies. Discrepancies will be documented by the Field Team Supervisor and will be made available to the Project Manager. The batch of samples will be given a laboratory number and each sample will be assigned a unique identification number. The sample custodian will be responsible for seeing that all samples are transferred to the proper analyst or stored in an appropriate secure area.

Laboratory personnel are responsible for the care and custody of samples from the time they are received or collected, until the time the sample is exhausted or returned to the custodian. The laboratories will maintain internal COC procedures consistent with their laboratory-specific Quality Assurance Manual included as described for ESC in Attachment A and for Speedie & Associates, Inc. in the applicable ASTM standards.

5.0 CALIBRATION PROCEDURES AND FREQUENCY

Standard calibration procedures exist for all field equipment to be used for on-site monitoring and testing. Laboratory equipment used for sample analysis also has prescribed calibration procedures. Any corrective action taken with respect to field equipment or laboratory instrumentation will be documented in either field notebooks or equipment maintenance records. These procedures, along with the required frequency of calibration, are discussed below.

5.1 FIELD INSTRUMENT CALIBRATION

The field equipment that will need calibration is listed below:

- photoionization detector

Proper maintenance, calibration and operation of each instrument will be the responsibility of field personnel assigned to a particular field activity. All instruments and equipment used during the field program will be maintained, calibrated, and operated according to the manufacturer's guidelines and recommendations. Calibration procedures for field equipment are summarized below. A schedule and record of instrument calibration will be maintained throughout the duration of the study. All field equipment requiring regular calibration will be calibrated at least once per day. Relevant manuals will be kept with field personnel during the performance of field activities. All equipment will receive routine maintenance checks to minimize equipment breakdown in the field or laboratory. Any items found to be inoperable will be taken out of use and a note stating the time and date of this action will be made in the field log.

5.2 FIELD CALIBRATION PROCEDURES

Field measurements may be collected using portable organic vapor monitor (OVM) that features VOC detection by photoionization detector (PID). The OVM will be used to measure organic gases and vapors in soil vapor as well as in ambient air.

With the OVM, manufacturer-supplied calibration standard span gas will be used to calibrate the meter. Calibration of the OVM will be performed once each day prior to use, and more frequently as needed throughout the day if irregularities in the readings become apparent.

Clear Creek will maintain a bound log book containing calibration data for each OVM, including time and date of the previous calibration, who performed the calibration, and how it was performed.

5.3 LABORATORY CALIBRATION PROCEDURES

Calibration of laboratory instruments is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established detection limits.

EPA Method 8260B: Each instrument is calibrated with standard solutions appropriate for the type of instrument and the linear range established for the analytical method. Each individual method contains requirements for the number and concentration of calibration standards, which are described in the laboratory's Quality Assurance Plan and Standard Operating Procedures. Each calibration is then verified through the use of statistical tests (e.g., a Pearson's Correlation Coefficient or relative standard deviation calculations), continuing calibration verification standards and blanks, Laboratory Control Samples, and Blank Spikes prior to the sample results being approved.

ASTMs Method D422, D854, D2216, and D2937: Each of the instruments used in these tests is calibrated according to standard practices, as outlined in the ASTM method protocols. Speedie & Associates, Inc. will calibrate each instrument as required by the relevant method.



6.0 DATA REDUCTION, REPORTING, REVIEW, AND QUALITY CONTROL

6.1 DATA REVIEW

Data review is the process of reviewing data and accepting, qualifying, or rejecting data on the basis of sound criteria. Iris and Clear Creek personnel will perform the data review. The data review will consist of a systematic review of the analytical results, associated QC methods and results, and all of the supporting data.

6.1.1 DATA REVIEW PROTOCOLS

Data review for groundwater analytical and soil geotechnical results will include a data completeness check of each data package, a transcription check for sample results, and a thorough review of all laboratory reporting forms and the associated raw data for QA/QC issues.

Specifically, this review will include the following for groundwater sample analysis:

- review of data package completeness;
- review of the required reporting summary forms and all associated raw data to determine if the QC requirements were met and to determine the effect of exceeded QC requirements on the precision, accuracy, and sensitivity of the data;
- review of raw data and all calculations associated with one VOC sample per sample delivery group (SDG) to determine if the sample results and quantitation limits were correctly calculated and reported;
- review of additional QA/QC parameters, such as: field duplicates and lab blank contamination, to determine technical usability of the data; and
- application of standard data quality qualifiers to the data, as described in the Arizona Department of Health Services (ADHS) *Arizona Data Qualifiers, Revision 2.0* and included in Attachment C.

This review will include the following for soil geotechnical analyses:

- review of data package completeness;

- review of the required reporting summary forms and all associated raw data to determine if the QC requirements were met and to determine the effect of exceeded QC requirements on the precision, accuracy, and sensitivity of the data;
- review of raw data and all calculations associated with one geotechnical sample per sample delivery group (SDG) to determine if the sample results were correctly calculated and reported. and

6.1.2 DATA REVIEW REPORT DELIVERABLES

The data review report deliverables for groundwater analysis will include the following information:

- a comprehensive narrative detailing all QC exceedances, explaining qualifications of data results. In cases where data are qualified due to quantifiable QC exceedances, the bias (high or low) will be identified. The narrative will provide an assessment of the usability of the data compared to the DQOs.

The data review report deliverables for geotechnical analyses will follow standard ASTM protocols.

6.1.3 DATA QUALIFIERS

ESC data qualifiers will be used to identify qualified groundwater analytical data and include them in the final data package for the groundwater samples.

6.2 QUALITY CONTROL REVIEW

During review of groundwater analytical data, results for QC sample analyses, including blanks and duplicates will be evaluated as described below, using the Precision, Accuracy Representativeness, Comparability, and Completeness PARCC QC criteria defined below, to determine the validity and usability of the data (EPA, 2002).

During review of geotechnical testing data, results will be evaluated using the QC guidelines provided in the ASTM standard methods.

6.2.1 PRECISION

Precision measures the reproducibility of repetitive measurements and is usually expressed in terms of imprecision. For this project, the precision of the groundwater analytical results will be evaluated by determining the relative percent difference (RPD) in results for the matrix

spike/matrix spike duplicate (MS/MSD) and field duplicate samples, using the following formula:

$$RPD = 100 \times \frac{(C_1 - C_2)}{(C_1 + C_2)/2}$$

Where:

RPD = relative percent difference
 C_1 = larger of the two observed values
 C_2 = smaller of the two observed values

Imprecision in MS/MSD results are usually an indication of sample matrix effects whereas imprecision of field duplicate (collocated) sample results may be an indication of sample heterogeneity or multiplicative interferences that diminish or enhance analytical signals. Sample results that do not meet precision objectives may still be considered usable for data quality objectives based upon professional judgment as to the cause and magnitude of the imprecision. All such decisions will be clearly justified in the data usability section of the review report.

6.2.2 ACCURACY

The determination of accuracy of a measurement requires knowledge of the true or accepted value for the signal being measured. It is a statistical measurement of correctness, and includes components of random error (variability due to imprecision) and systematic error (bias). It therefore reflects the total error associated with a measurement. Errors as they pertain to accuracy may result from several sources including the sampling processes, field contamination, improper preservation, handling of samples, sample matrix biases, and analytical procedures. A measurement is accurate when the reported value does not differ from the true value, or known concentration of the spike or standard. Accuracy of groundwater analytical data for this project will be calculated in terms of percent recovery of a known amount of a spiked compound according to the following formula:

$$\%R = 100 \times \frac{(C_S - C_N)}{C_{SA}}$$

Where:

%R = percent recovery
 C_S = measured concentration in spiked aliquot
 C_N = measured concentration in unspiked aliquot
 C_{SA} = actual concentration of spike added

Accuracy will be evaluated based upon percent recoveries of the internal standards, MSs, MSDs, system monitoring compounds, and surrogates for VOC analyses. The following are specific guidelines for the usability assessment of QC results that give an indication of the accuracy of the sample data.

Reference or Check Standard Evaluation. Standard reference materials analyzed by the laboratory following EPA method protocols (e.g. laboratory control sample [LCS], calibration check samples [CC]) are compared with true values and acceptable ranges as indicators of accuracy. Values outside the acceptable ranges require corrective action to determine the source of error and provide for implementation of a corrective action. Affected sample results may be considered estimated or unusable during usability assessment.

Surrogate Standard Evaluation. The results of surrogate standard determinations are compared with the true values spiked into the sample matrix before extraction and analysis, and the percent recoveries of the surrogate standards are determined. If these recoveries fall outside of the control limits, the associated data may be affected. During the usability assessment, results may be considered as estimated or unusable based on the surrogate recoveries.

Matrix Spike Evaluation. The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy, as defined by the percent recovery. If the accuracy value exceeds the acceptance criteria for the given compounds, the sample set may need to be reanalyzed for the parameter in question, or the results considered as estimated.

Additionally, blanks will be used to evaluate whether laboratory procedures represent a possible source of contamination in the field samples. Unmonitored contamination can allow false positive results to be reported and treated as true sample components, when in fact they are not; this type of error will adversely affect the accuracy of the reported results. Method blanks will be used to evaluate contamination for volatile organic analyses for this project.

For the laboratory, method blanks for the volatile analyses have specific criteria that must be met for compliance. The general procedures for assessing blank samples are as follows:

1. Tabulate the target compound results for all blank samples.
2. Identify all blank samples for which target compounds are reported above the required reporting limit following EPA guidelines.
3. If no compounds are detected in any of the blank samples, the data are reported unqualified for blank contamination.

4. If any compounds are found in any of the blank samples, their concentration(s) will be reported in the data review and assessed. Appropriate qualifiers will be added to the data summary tables in the review reports.

If an analyte is detected in a method blank(s), a blank action level will be calculated as five times the highest blank concentration for all samples associated with that method blank. Common laboratory contaminants, such as acetone, methylene chloride, 2-butanone, and toluene, will have a blank action level of 10 times the amount in any associated blank.

Blank actions may raise the sample-specific reporting limits to levels greater than the project-required levels. All determinations of usability for accuracy will be justified in the usability section of the data review report.

6.2.3 REPRESENTATIVENESS

Representativeness expresses the degree to which the sample data accurately and precisely represent the media being sampled at a specific location at a specific time. Representativeness will be achieved, in part, through use of the standard and analytical procedures described in this QAPP. Sampling locations shall be chosen carefully and a sufficient number of investigative samples will be collected to maintain site representation. Method blanks are QC samples used to help differentiate between laboratory constituents, and the constituents actually representative of site groundwater. One measure of representativeness is field duplicate precision. If field duplicate precision criteria are not met, the data reviewer will determine usability of the associated results and qualify the data based upon best professional judgment. Poor field duplicate precision may be an indication of sample heterogeneity or poor sampling.

6.2.4 COMPARABILITY

Comparability is the confidence with which one data set can be compared to other data. The objectives for this program are to produce data with the greatest degree of comparability possible. Comparability will be achieved by following procedures outlined in the QAPP, including using standard methods for sampling and analysis, reporting data in standard units, and using standard and comprehensive reporting formats.

6.2.5 COMPLETENESS

Measurements of completeness can be defined as the ratio of usable measurements obtained to the total number of planned measurements for an activity. Completeness can be defined as:

$$\% \text{Completeness} = 100 \times \frac{\text{number of valid results}}{\text{number of expected results}}$$

Additionally, for a data set to be considered complete, the data set must contain all QC check analyses verifying precision, accuracy, and sensitivity for the analytical protocols. This will be determined during the data review process. Following data review and usability assessment, the percent completeness can be calculated.

6.2.6 SENSITIVITY – DETECTION LIMITS

The groundwater analytical method detection limit (MDL) is the minimum concentration of an analyte that can be identified using a specific method. The laboratory usually determines the MDL by calculating the standard deviation of the results of seven replicate spike sample analyses and multiplying by three, using reagent water as a sample. The MDL is an ideal detection limit when there is no background laboratory contamination and the sample to be analyzed is a clean sample free of matrix effects. MDLs, also known as the method quantitation limit, are determined using reagent water as the sample. MDLs are not field sample specific and do not vary with the sample preparation or dilutions required for each field sample analyzed. The MDL is the instrument detection limit (IDL) plus adjustments for typical sample preparation techniques.

Sensitivity for the groundwater analytical data for this project will be evaluated against the specific project MDLs and internal standard recoveries, as indicators of gas chromatograph/mass spectrometry (GC/MS) sensitivity. Quantitation limits may be affected by matrix interferences, such as those caused by samples with high concentrations of VOCs. If a dilution is required, the lowest dilution necessary to bring the compound within range should be performed in all cases. In cases where the specified detection limits (or project-specific reporting limits) are not achieved, the usability of the data will be evaluated by the Independent Data Validator in conjunction with the Project Manager and Project QA Coordinator.

For the groundwater samples, the laboratory will report all data to the laboratory reporting limit. Laboratory reporting limits for EPA Method 8260B for ESC are at least twice the MDL and are listed in Attachment A.

The laboratory reporting limit is sample dependent and may vary from sample to sample, depending on the sample size, matrix interferences, moisture content (soil), dilution factors, and other sample- or matrix-specific variables. The laboratory must qualify data below their standardized reporting or quantitation limits.

7.0 DATA MANAGEMENT PLAN

7.1 FIELD DATA

The Field Team Supervisor will forward field data records and copies of the Chain-of-Custody (Attachment D) to the project office at the conclusion of each sampling day.

7.2 LABORATORY DATA

Final analytical laboratory results will be provided in both hard copy and electronic data deliverable (EDD) format.

7.3 DATA MANAGEMENT

Water level and analytical water quality data will be maintained in a Microsoft Excel or Access compatible format.

8.0 QUALITY CONTROL REQUIREMENTS

The principal goal of any sampling and analysis program is to obtain accurate and representative environmental samples and to provide valid analytical data. QC measures are implemented so that the accuracy and representativeness of field and analytical data can be evaluated and documented. The quality of the field data will be determined on a regularly scheduled basis through the use of field QC samples. Laboratory QC samples will also be analyzed according to reference analytical method protocols to ensure that laboratory procedures and analyses are properly performed.

For this project, the types of QC samples collected and analyzed will be defined and their roles in the assurance of the production of QC data will be discussed in the following sections. Additional QC procedures not discussed in this section should also be implemented by field and laboratory personnel according to specific method protocols and the Work Plan. These additional QC procedures also serve to ensure the quality and reproducibility of the analytical data.

8.1 FIELD QUALITY CONTROL SAMPLES

Field groundwater QC samples are collected for laboratory analysis to check sampling and analytical precision, accuracy, and representativeness. The three types of field samples to be used as QC check samples are field blanks, trip blanks, and field duplicates. Each field blank and duplicate QC sample will be assigned a unique number so that the laboratory will not know which samples are field blanks or duplicates. Trip blanks will be prepared by the laboratory and identified as such. Field blank and duplicate QC samples will be identified in the field activities logbook and/or on sampling information forms according to type.

There are no field quality control samples collected for geotechnical soil analyses.

8.2 LABORATORY QUALITY CONTROL SAMPLES

Laboratory QC includes the daily specific measurements taken within the laboratory to verify sample integrity, performance of analysis, data processing, and record maintenance. QC is the mechanism established to control errors. In addition to QC samples collected in the field, the laboratory will analyze QC samples as required by the analytical methods to ensure analytical precision, accuracy, and representativeness.

For groundwater analyses, all positive sample results will be confirmed by either GC/MS or analysis on a second column as described in the referenced method, and samples and laboratory QC samples will be analyzed in accordance with the QC acceptance criteria.

Typical groundwater analytical laboratory QC samples include blank spikes, Laboratory Control Samples (LCSs), method blanks, surrogates, matrix spike/matrix spike duplicate (MS/MSD) analysis, internal standards, duplicate sample analysis, and known value check standard analysis. The spiking compounds and concentrations are in accordance with method requirements and are described in ESC's Quality Assurance Plan and Standard Operating Procedures (Attachment A).

Speedie & Associates, Inc. will follow standard ASTM method guidelines for geotechnical analytical laboratory QC samples.



9.0 PERFORMANCE AND SYSTEM AUDITS

The Project QA Coordinator will monitor the performance of the field and laboratory quality assurance program. This will be achieved through regular contact with field and laboratory personnel. Upon identification of a problem and the need for corrective action, the Project QA Coordinator may initiate an audit.

9.1 FIELD PERFORMANCE

Field performance will be monitored through review of sample collection documentation, sample handling records (COC forms), field notes, and field measurements and by unannounced field operations audits conducted by the Project QA Coordinator or the Project Manager. The Project QA Coordinator will report the audit findings and any need for corrective action to the Project Manager. The Project Manager will be responsible for supplying the audit findings to the appropriate personnel.

9.2 LABORATORY PERFORMANCE AUDITS

The Project QA Coordinator will be in contact with the analytical and geotechnical laboratories while samples collected during this investigation are being analyzed. This will allow assessment of progress toward obtaining data quality objectives and the ability to make corrective measures as problems arise.

10.0 PREVENTATIVE MAINTENANCE

Regularly scheduled preventive maintenance will be performed to keep all field and laboratory equipment in good working condition.

10.1 FIELD

Preventative maintenance of field measurement instrumentation and equipment will be performed according to manufacturer's instructions. The field staff is responsible for ensuring that all instrumentation is operating properly prior to use. If problems are encountered, they will be documented in the field notes. The faulty instrumentation or equipment will be scheduled for repair and sequestered and tagged until repaired and qualified for re-use.

10.2 LABORATORY

Preventative maintenance of analytical instrumentation and equipment within the laboratory will be the responsibility of the laboratory personnel and analysts and will be performed as outlined in ESC's Quality Assurance Manual included in Attachment A. This maintenance in a groundwater analytical laboratory includes routine care and cleaning of instruments and inspection and monitoring of carrier gases, solvents, and glassware used in analyses. Speedie & Associates, Inc. will maintain geotechnical laboratory equipment according to standard ASTM method requirements. Maintenance logs will be employed to document routine maintenance, problems, and repairs.

Precision and accuracy data are examined for trends and excursion beyond control limits to determine evidence of instrument malfunction. Maintenance will be performed when an instrument begins to change as indicated by the degradation of peak resolution, shift in calibration curves, decrease in sensitivity, or failure to meet one or another of the QC criteria.

Maintenance not only applies to major analytical instrumentation but also to other laboratory equipment such as refrigerators, thermometers, and balances that can have an impact on data quality if in need of repair or calibration.

11.0 CORRECTIVE ACTIONS

If QC audits result in detection of unacceptable conditions or data, the Project Manager, in conjunction with the Project QA Officer, will be responsible for developing and directing implementation of corrective actions. Corrective actions will include one or more of the following:

- identifying the source of the violation
- evaluating and amending sampling and analytical procedures
- accepting data and flagging the data to indicate the level of uncertainty associated with failure to meet the specified QC performance criteria

Any finding requiring corrective action must be documented to the Project Manager. This documentation will be supplied to the appropriate personnel by the Project Manager. The Project QA Coordinator will check to ensure that corrective actions have been implemented and that the problem has been resolved. More easily addressed problems may be encountered in the field or the laboratory. Such problems will be addressed and the corrective action noted in the appropriate lab or field notebook.

12.0 QUALITY ASSURANCE REPORTS

The Project QA Coordinator will keep the Project Manager informed of quality issues such as audit reports, problems, and corrective actions, and other applicable issues. Verbal reports will be presented as issues arise, followed by timely written documentation. A QA/QC progress report will be written and presented to management on a quarterly basis.

The laboratories and field teams will be routinely provided copies of laboratory and field data and data review reports. These data and reports will be reviewed and the Project QA Coordinator and Project Manager will be kept informed regarding pertinent issues.

13.0 REFERENCES

Arizona Department of Health Services (ADHS). 2003. *Arizona Data Qualifiers, Revision 2.0.* November 26, 2003.

Clear Creek Associates and Iris Environmental. 2011. RCRA Facility Investigation (RFI) Work Plan. Clear Creek Associates and Iris Environmental, December 20, 2011.

United States Environmental Protection Agency (EPA QA/R-5), *EPA Requirements for Quality Assurance Project Plans*, March 2001 (USEPA 2001)

United States Environmental Protection Agency (EPA QA/G-4), *Guidance for the Data Quality Objectives Process*, February 2006 (USEPA 2006)

United States Environmental Protection Agency (EPA QA/G-5). *EPA Guidance for Quality Assurance Project Plans, (EPA QA/G-5)*. December 2002.

TABLES

Table 1 – Data Quality Objectives
Former Romic Environmental Technologies
RFI Workplan
Gila River Indian Community, Arizona

STEP 1	STEP 2	STEP 3	STEP 4	STEP 5	STEP 6	STEP 7
State the Problem	Identify the Goal	Identify Inputs to the Decisions	Define Study Boundaries	Develop Decision Rules	Specify Tolerable Limits on Errors	Optimize Sampling Design
<p>The Romic facility is a former hazardous waste treatment and recycling facility located on the Lone Butte Industrial Park leased from the Gila River Indian Community (Site). Beginning in 1975, the prior owner of the facility accepted a variety of hazardous materials for recycling or treatment. Romic assumed operations in 1988. The facility was reconstructed between 1988 and 1991, which included remediation of shallow soils and installation of a liner beneath units. Romic continued operations into 2007. RCRA Closure was granted in March 2010.</p> <p>VOCs were detected in 2008 in shallow soil gas samples collected between 5 and 15 ft bgs and in deep soil gas samples collected above the perched groundwater zone, between 15 and 47 feet bgs. VOCs were mostly removed from soils via soil vapor extraction.</p> <p>Groundwater monitoring in on- and offsite wells plus a vertical VOC sampling profile detected chlorinated VOCs at the water table of the regional aquifer. No VOCs were detected at depth (below 165 ft bgs) in the regional aquifer. Low concentrations of VOCs and acetone were detected in the perched groundwater zone between 40 and 60 ft bgs.</p> <p>A groundwater monitoring program has continued and expanded since 2007 to determine the vertical extent of VOCs. The plume has been fully defined at the site and mostly offsite. Downgradient, vertical characterization is not complete in distal end.</p>	<ol style="list-style-type: none"> Evaluate offsite vertical limit of the VOC plume by determining the vertical profile of VOC concentrations within the regional aquifer. Evaluate whether VOC plume is present beneath well screens in existing, distal wells. Evaluate need for the installation of an additional groundwater monitoring well south of LB-14. Evaluate VOC mass between LB-4 and LB-5. Evaluate risk due to concentrations of VOCs in soil gas by site-specific modeling using geological characteristics of shallow vadose zone. 	<ul style="list-style-type: none"> Depth-specific groundwater samples from the regional groundwater zone (below 70 feet bgs) analyzed for VOCs by USEPA Method 8260B at a fixed lab (Goal 1 through 4). Shallow geotechnical soil samples analyzed for Soil Bulk Density by ASTM D2937, Grain Density by ASTM D854, Soil Moisture Content by ASTM D2216, and Particle Size Analysis by ASTM D422 at a fixed lab (Goal 5). Low laboratory reporting limits (RLs) sufficient to compare concentrations to screening criteria (Goals 1 through 5). 	<ul style="list-style-type: none"> The study boundaries include the Site and the plume boundaries to the west. Groundwater samples will be collected from the regional aquifer (75 to 140 feet bgs). Within the time frame of initial field work, the location and installation of additional monitoring well(s) may vary depending on the results of the groundwater analytical results. 	<ul style="list-style-type: none"> If concentrations of VOCs in groundwater from the regional aquifer are not detected above laboratory RLs, then additional investigation of regional aquifer may not be warranted. If VOCs in groundwater from the regional aquifer are detected above laboratory RLs in multiple samples at depths and locations consistent with conceptual model, then additional characterization may not be warranted and no additional monitoring wells required. If characteristics of the vadose zone are below vapor intrusion risk levels, mitigation may not be warranted. 	<p>Sampling error and measurement error are associated with environmental data collection and may lead to decision errors. Sampling error occurs when the sample is not representative of the true condition of the environment at the site. Measurement error occurs because of random and systematic errors associated with sample collection, handling, preparation, analysis, data reduction, and data handling. Decision errors are controlled by adopting a scientific approach that uses hypothesis testing to minimize the potential for error.</p> <p>There are two types of decision error: false negative error and false positive error. A false negative decision error occurs when the null hypothesis is rejected, although it is true. The consequences of a false negative error would be that contaminated media that pose an unacceptable risk to potential human or ecological receptors at the site are not addressed. A false positive decision error occurs when the null hypothesis is not rejected, although it is false. The consequences of a false positive error are that unnecessary resources are expended on further action to address contaminated media that did not exist at levels that would exceed action levels or acceptable risk levels.</p> <p>Tolerable limits on sampling decision errors cannot be precisely defined; however, the decision errors will be minimized by evaluating the potential source areas. For this investigation, sampling locations are in suspected potential source areas. Decision errors based on analytical data will be minimized by the use of USEPA-approved analytical methods.</p> <p>Data quality will be evaluated using measurement quality objectives as specified in the QAPP.</p>	<p>Four borings will be drilled offsite. Depth-specific groundwater samples will be collected from each boring to characterize the regional groundwater aquifer. Therefore, all samples are located in areas that will yield representative data that are necessary to make sound decisions and recommendations.</p> <p>Groundwater samples will be collected using standard methodology and equipment. The analytical method selected for groundwater samples can detect potential contaminants of concern at concentrations above most screening level guidance. Analytical methods chosen are all USEPA approved and are commonly implemented. Sample collection methods and analytical methods are discussed in the Work Plan and QAPP.</p> <p>A geotechnical soil sample will be collected using standard methodology and equipment. The analytical method selected for geotechnical analysis can characterize soils to the degree of detail needed for vapor intrusion risk evaluations. Analytical methods chosen are all ASTM/USEPA approved and are commonly implemented. Sample collection methods and analytical methods are discussed in the Work Plan and QAPP.</p>

Notes:

bgs	Below ground surface
QAPP	Quality Assurance Project Plan
RL	Reporting Limits
USEPA	U.S. Environmental Protection Agency
VOC	Volatile organic compounds

Table 2 – Sample Containers and Preservation

Former Romic Environmental Technologies

RFI Workplan

Gila River Indian Community, Arizona

Analysis	Container		Preservative (chill to 4° C)		Maximum Holding Time (from sampling date)	
	Water	Soil	Water	Soil	Water	Soil
Organic Chemistry						
EPA 8260B	(3) 40ml VOAs (headspace free)	NA	Chill to 4° C 0.5 ml HCl pH < 2	NA	14 days	NA
Geotechnical						
ASTM D422	NA	Stainless steel tube	NA	None	NA	None
ASTM D854	NA	Stainless steel tube	NA	None	NA	None
ASTM D2216	NA	Stainless steel tube	NA	None	NA	None
ASTM D2937	NA	Stainless steel tube	NA	None	NA	None

Notes:

ASTM = American Society for Testing and Materials

C = Celsius

EPA = Environmental Protection Agency

HCl = Hydrochloric acid

ml = millileter

NA = not applicable

VOAs = Volatile Organic Analysis

ATTACHMENT A

ENVIRONMENTAL SCIENCE CORP. STANDARD OPERATING PROCEDURES



ENVIRONMENTAL SCIENCE CORPORATION
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SOP Revision Summary

SOP:			
Author - Michael Jacobs	Number - 330363	Department - Volatiles	
Title - VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)			
Revision - R16	Rev. Date - 01/30/2009		

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
1	07/30/2009	7.4.1, 7.4.2, 8.2.3.1, 8.2.3.2, 8.2.3.3	Addition of Oxygenates calibration and QC requirements
		8.2.6.4 & 9.11	Addition of South Carolina Requirements
2	09/03/2009	9.11	Addition of equal weighting or 1/x regression
3	09/18/2009	11.11	Addition of client note
4	10/02/2009	Varous	Addition of 8260C requirements
5	10/30/2009	8.3.4	Added to list of poor performers
6	10/07/2009	11.7.1	Correction for marginal excedences

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(see Revision History at the end of this document for more information)

TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)

SOP NUMBER: 330363

PREPARED BY: Michael Jacobs

REVIEWED BY: Ben Wilson/Dixie Marlin

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Department Manager

QA Department

1.0 SCOPE AND APPLICATION

- 1.1 This standard operating procedure is used to determine volatile organic compounds in a variety of matrices. This SOP is designed for EPA methods 8260B, 624, 6200B, KSGRO, or similar volatile GC/MS analyses. This procedure is applicable to nearly all kinds of samples, regardless of water content, including ground water, aqueous sludge, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments. The compounds that can be determined using this SOP are listed in Attachment III, which contains a list of the typical primary and secondary ions used in determining these compounds.
- 1.2 Reporting Limits (RLs) are listed in the following Table 1. Compounds routinely analyzed by this method and their typical reporting limits are included in the following table (subject to change).

Table 1 - 8260/624/6200B Reporting Limits and Common Calibration List

Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
Acetone	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Acrolein	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Acrylonitrile	0.010	mg/L	0.010	mg/Kg	0.5	mg/Kg
Benzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromodichloromethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromoform	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg

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<u>Compound</u>	<u>RL</u>	<u>Units</u>	<u>RL*</u>	<u>Units</u>	<u>RL</u>	<u>Units</u>
Bromomethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
n-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
sec-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
tert-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Carbon tetrachloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorodibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
2-Chloroethyl vinyl ether	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Chloroform	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Chloromethane	0.0025	mg/L	0.001	mg/Kg	0.05	mg/Kg
2-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dibromo-3-Chloropropane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2-Dibromoethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,4-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dichlorodifluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,1-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
cis-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
trans-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
cis-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
trans-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Ethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexachlorobutadiene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexane	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
Isopropylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
p-Isopropyltoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2,2,4-Trimethyl Pentane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)

Compound	RL	Units	RL*	Units	RL	Units
2-Butanone (MEK)	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Methylene Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
4-Methyl-2-pantanone (MIBK)	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
Methyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Naphthalene	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
n-Propylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Styrene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,1,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Tetrachloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Toluene	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2,3-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,1-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2,3-Trichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3,5-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Vinyl chloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Xylenes, Total	0.003	mg/L	0.003	mg/Kg	0.15	mg/Kg
Additional Compounds						
Acetonitrile	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Allyl Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Chloroprene	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Trans-1,4-Dichloro-2-butene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
Isobutanol	0.100	mg/L	0.100	mg/Kg	5.0	mg/Kg
1,4-Dioxane+	0.100	mg/L	0.100	mg/Kg	5.0	mg/Kg
Methacrylonitrile	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Methyl Methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Ethyl methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Propionitrile	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Pentachloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Carbon Disulfide	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Cyclohexanone	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
2-Hexanone	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
Iodomethane	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)

Compound	RL	Units	RL*	Units	RL	Units
Isobutanol	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Propionitrile	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Vinyl Acetate	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Tetrahydrofuran	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Bromoethane	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
2-Butanol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Ethanol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Tert-Butyl Alcohol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Ethyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Methyl-tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Tert-Butyl alcohol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Tert-Amyl Methyl Ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Tert-Butyl Formate	0.020	mg/L	0.020	mg/Kg	1.0	mg/Kg
Tert Butyl Ethyl Alcohol	0.100	mg/L	0.100	mg/Kg	5.0	mg/Kg
Tert Amyl Alcohol	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Dichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg

RLs are based on a 5mL purge volume

Low Soil - Using a 5g soil sample to 5mL water – See Method 5035 (SOP# 330751) Section 8.2.4.1

High Soil – Using 100uL extract from 5g. soil sample to 5mL methanol – See Method 5035 (SOP# 330751) Section 8.3.1.2

- + 1-4,Dioxane has a RL of .004 when run using the SIM mode.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 Volatile organic compounds (VOCs) are determined from a 5mL sample withdrawn from a sealed 40mL vial. For water samples analyzed for low levels of analytes using Method 5030 (SOP 330752), the entire vial is placed into the instrument autosampler. The autosampler purges 5mL of sample and adds 1µL of surrogate standards and internal standards. An inert gas is bubbled through a sparger needle inserted into the sample. The purged volatile components then travel via a transfer line to a sorbent trap. When purging is complete, the trap is rapidly heated. The trap is backflushed with a helium carrier gas, to transport the desorbed sample components into a gas chromatographic (GC) column. The GC column separates and carries the components to a mass spectrometer (MS) or a specific detector, depending on the determinative method selected.
- 2.2 For other samples, Method 5035 (SOP 330751), volatile organic compounds are determined from a 5g sample combined with 5mL reagent water.

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- 2.3 Initial/Continuing Calibration Verification (ICV/CCV) – Analytical standard run at the beginning of every 12 hours shift to verify the stability of the established initial calibration of the analytical system.
- 2.4 Continuing Demonstration of Capability (CDOC) – At least, annual verification of analyst continued ability to perform method acceptably.
- 2.5 Duplicate – A second aliquot of a field sample analyzed using identical preparation and analytical procedures. Analysis of a sample duplicate monitors precision associated with laboratory procedures.
- 2.6 Initial Demonstration of Capability (IDOC) - A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. See also Continuing Demonstration of Capability (CDOC).
- 2.7 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) – Duplicate aliquots of a control sample of known in composition. This sample is prepared from a source that is different from the stock used to prepare the initial and continuing calibration standards. LCS/LCSD are analyzed exactly like a sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Method precision can be determined using the results of the LCS/LCSD analysis.
- 2.8 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.
- 2.9 Method Blank - An analytical control consisting of all reagents used in the analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.
- 2.10 Method Detection Limit - The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero.
- 2.11 Reporting Limit (RL) – Also see Practical Quantitation Limit (PQL). Routinely the reporting limit is the lowest standard of the calibration curve. Technically, the reporting limit is the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions.

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- 2.12 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the detection limit.
- 2.13 Second Source Calibration Verification (SSCV) – A mid-point or low standard made from a secondary standard that is not used to construct the calibration curve. The SSCV is used to represent the calibration accuracy of the instrument and must perform within method stated criteria.
- 2.14 Surrogate - A compound which is similar to the target analytes in chemical composition and behavior and not expected to occur naturally in field samples that is spiked by preparation/analytical personnel to assess sample preparation and analytical efficiency in each individual field sample.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Specific cautions are included for known extremely hazardous materials.
- 3.2 For specific information regarding the toxicity of the chemicals used in this method, a reference file of Material Safety Data Sheets (MSDS) is made available to all staff.
- 3.3 Safety glasses, gloves, and a laboratory coat must be worn to protect against physical contact with samples and laboratory reagents that contain potentially hazardous chemicals.
- 3.4 Waste generated from the procedure need to be disposed according to ESC policy and local, state, and federal law.
- 3.5 Glycol ethers are suspected carcinogens. All solvent handling should be performed in a hood while using proper protective equipment to minimize exposure to liquid and vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 Volatile analysis for water and sodium bisulfate preserved soil samples must be completed within 14 days from the time of sample collection. Water samples that are not chemically preserved must be analyzed within 7 days. It is also an ESC requirement that water samples with 2-chloroethylvinyl ether (2-CEVE), as a compound of interest, be collected unpreserved and analyzed within 7 days of collection. It has been shown that the acid preservative reacts with the 2-CEVE, which could result in false negative reporting of 2-CEVE in samples. Unpreserved soil samples must be analyzed within 48

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hrs from the time of collection or otherwise frozen at -12+/-3°C. High-level soil samples collected in Encore™ or equivalent type sampling devices must be placed in vials of methanol according to Method 5035 (SOP# 330751).

STATE NOTE: The State of South Carolina and the OHIO VAP requires that all soil samples must be collected and analyzed using Method 5035. Samples must be preserved within 48 hours from the time of collection, if collected in Encore™ type sampling devices. The holding time for soil samples preserved with methanol or sodium bisulfate is 14 days from the time of collection.

- 4.2 Aqueous samples must be collected in at least duplicate in 40mL vials with 0.008% Na₂S₂O₃ per liter if residual chlorine is present. Sample kits can be configured to request additional vials per client request. The pH must be adjusted to <2 with HCl. Soil samples must be collected by one of the following: 1) A 4oz. soil jar filled with soil with zero headspace, 2) Two 5g samples preserved in the field with 5mL NaHSO₄ to a pH<2 and one 5g sample preserved in the field with methanol (for high level analysis) or 3) A 5g or 25g sample collected in an Encore or equivalent type sampling device and frozen in the laboratory within 48 hours from the time of collection.

For all soil samples, a 40z soil jar should also be collected to determine percent solids. All samples and extracts must be shipped and stored at 4°C.

STATE NOTE: Soil and Water samples received from the states of Missouri or Kansas may be preserved with tri-sodium phosphate and will have a resulting pH of 14.

STATE NOTE: For Ohio VAP samples, Encore samplers must be collected in the field and shipped to the laboratory to be frozen within 48 hours of collection. These samples will remain frozen until analysis and do not require additional chemical preservation. The holding time for these samples remains at 14 days from the time of collection. The holding time begins at collection and ends at 14 days.

5.0 INTERFERENCES

- 5.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components must be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.
- 5.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the

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purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted.

- 5.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations, one or more instrument blanks must be analyzed to check for cross contamination.
- 5.3.1 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.
- 5.3.2 For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C.
- 5.3.3 In extreme situations, the whole purge and trap device may require dismantling and cleaning.
- 5.3.4 Screening of the samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique. by SW-846 Method 3820, Hexadecane Extraction and Screening of Purgeable Organics), or screening of 5mL of sample using an HNU or equivalent portable PID.
- 5.4 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst must be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.
- 5.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. A storage blank must be analyzed weekly to check for cross contamination of samples while samples are stored in the volatiles laboratory walk-in cooler. The storage blank is prepared from organic-free water and is placed in the cooler for a period of one week. After a week, it is analyzed to verify that no contamination of client samples has taken

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place due to contamination in the storage unit. Analysis of a storage blank must be performed for both the volatiles laboratory walk-in coolers.

- 5.6 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200° C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately 50 times higher due to poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.
- 5.7 Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If a large amount of effervescent gas is generated, the sample may lose a significant amount of volatile analytes. If a sample effervesces, an unpreserved sample will be collected to eliminate volatiles loss whenever possible. The holding time for unpreserved VOC samples is seven days, rather than 14 days.
- 5.8 An analyst may re-analyze any sample if instrumentation or human error is suspected. This includes all QC samples, which can only be re-analyzed twice. If failure continues, instrument maintenance must be performed and/or the instrument must be re-calibrated.
- 5.9 Glassware must be scrupulously cleaned. All glassware must be cleaned per EPA protocol, as stated in SOP # 030701, *Glassware Cleaning*.

6.0 EQUIPMENT AND SUPPLIES

The operation, cleaning, and scheduled maintenance procedures, as prescribed by the equipment manufacturer, are followed as provided in the Operator's Manuals. Documentation of maintenance or system modifications are recorded in a maintenance logbook which accompanies each analytical system.

- 6.1 Instrumentation: All instrumentation meets or exceeds EPA method requirements.
 - Use (method #'s): 8260, 624
 - Model #: HP 5890, or equivalent
 - Column (type, brand, size): J & W Scientific DB VRX 75 m x 0.450 mm, 2.55 micron, or equivalent.
 - Detector: MS 5972 or better.
 - Software name and version: HP Chemstation G1701BA B.01.00, or equivalent
 - Sample introduction system: Archon Autosampler, Encon P & T, or equivalent
- 6.2 Glassware must be scrupulously cleaned. All glassware must be cleaned per EPA protocol, as stated in SOP # 030701, *Glassware Cleaning*. Rinsing with methanol and laboratory reagent water cleans the volumetric flasks and graduated cylinders. The

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volumetric flasks are dried in a low temperature oven at less than 120°C and never cleaned with a brush or strong alkali solution.

- 6.3 The carrier gas used for volatiles analysis is Helium-5.0 grade.
- 6.4 Syringes used for preparing the calibration curve and preparing samples and sample dilutions are Hamilton brand (or equivalent). Syringe sizes used are 0.50µL, 10µL, 25µL, 50µL, 100µL, 250µL, 1mL, and 5mL.
- 6.5 Glass Sample (VOA) and Standard Vials:
 - 6.5.1 40mL VOA vials with a Teflon™/silicone septa and polypropylene open-top cap.
 - 6.5.2 8mL vials with Teflon™/silicone/Teflon™ septa and polypropylene open-top cap. (Used to store unused standards).
- 6.6 Miscellaneous:
 - 6.6.1 Stainless Steel Spatula or wooden tongue depressor.
 - 6.6.2 Disposable aluminum drying dishes - VWR #25433-008, or equivalent
 - 6.6.3 Teflon™-coated stir bars, 8mm x 16mm
 - 6.6.4 Laboratory Sand: Sand is prepared by rinsing clean, white sand with methanol and laboratory reagent water several times. The rinsed sand is baked in an oven at 175°C overnight to remove any volatiles and is then stored in the same oven. The heated laboratory sand is occasionally purged with carrier grade helium or nitrogen to remove trapped volatiles.
- 6.7 Oven: Fisher IsoTemp Forced-Air Oven with capabilities of 100°C, or equivalent
- 6.8 Top-loading Balance, capable of weighing to 0.01g, or equivalent.

7.0 REAGENTS AND STANDARDS

NOTE: All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system.

- 7.1 Laboratory water created by reverse osmosis/DI filtration evaluated to .055uS/cm to ensure purity. Laboratory water is used in all blanks to assure that it contains less than the minimum detection limit (MDL) of all compounds of interest. The blank must be assessed to ensure that the water does not show any detection of any VOC compounds.

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If volatile compounds are detected in the blank above the MDL all samples associated with this blank must be flagged.

- 7.2 Methanol, CH₃OH – purge-and-trap grade, demonstrated to be free of analytes. Store apart from other solvents.
- 7.3 Sodium Bisulfate, Na₂S₂O₃ - QEC Level 3 Certified, or equivalent
- 7.4 STOCK SOLUTIONS – Calibration and LCS
 - Stock calibration solutions must be purchased as certified solutions.
 - Certificates must be kept on file.
 - All Stock standards must be stored below -10°C
 - All non-gas stock standards must be replaced after six months, or sooner, if check standards indicate a problem.
 - Both gas and liquid standards must be monitored closely by comparison to the initial calibration curve and by comparison to a second source initial calibration verification standard.
 - Gas intermediate/secondary standards must be replaced weekly, or sooner, if comparison to check standards indicates a problem.
 - Non-gas intermediate/secondary standards must be replaced after six months, or sooner, if comparison to check standards indicates a problem.
- 7.4.1 CALIBRATION - Primary source standards is used to prepare the initial 5-point calibration curve (additional levels may be used as needed), the initial calibration verification (ICV), and continuing calibration verification (CCV) standard. The ICV and CCV are analyzed to verify the initial calibration and are prepared using the primary source standards used to produce the calibration curve. See Section 8.2.3.1 or 8.2.3.2 for the instrument preparation of the calibration standards. When primary standards are consumed, new standards must meet the same QC criteria as the consumed standards. Stock standards must be stored below -10°C and have a six-month holding time once opened. The expiration date of the diluted standards must not exceed the expiration date of the stock standards from which they are prepared. Once diluted, the standard must be replaced weekly.

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ICV Mix

The ICV solution is prepared in methanol in a 50mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	8260 Custom Mix 1	Q-4146	5	50
NSI	8260 Custom Mix 2	Q-4147	5	250
Absolute	n-Hexane	70962	2.5	50
NSI	1+2 MethylNaphthalenes	Q-4260A	2.5	50
NSI	Acrolein	Q-3835	2.5	250
NSI	8260 Gases Mix	CUS-5661	25	50

GRO ICV Mix

The GRO ICV solution is prepared in methanol in a 10mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Restek	TX TPH matrix spike mix	Cat 31484	1	1000

7.4.1.1 For soil/water autosamplers (5mL), a dilution of 10X is required for the ICV mix.

The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is below -10° C.

7.4.2 LABORATORY CONTROL STANDARDS - The standard list of target LCS compounds are those compounds listed in TABLE 11.7. Secondary source standards must be used to prepare the laboratory control standard (LCS) and the matrix spike and their duplicates. These standards are purchased from a different vendor or the primary vendor can supply different lot numbers, if a separate vendor is not available. The standard is at a concentration near the mid-level calibration standard. Stock standards must be stored at or below -10°C and have a six month holding time once opened, except the ICV gases which have 1 week holding time. Once diluted, the standard must be replaced weekly.

LCSs & Spikes

Prepare the LCS/LCSD in methanol in a 50mL volumetric flask as follows. A separate source or separate lot number is used for standard verification. The standard list of target LCS compounds are those compounds listed in TABLE 11.7.

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Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	8260 Custom Mix 1	Q-4146	5	50
NSI	8260 Custom Mix 2	Q-4147	5	250
Absolute	n-Hexane	70962	2.5	50
NSI	1+2 Methylnaphthalenes	Q-4260A	2.5	50
NSI	Acrolein	Q-3835	2.5	250
NSI	8260 Gases Mix	CUS-5661	25	50

GRO LCS

The GRO LCS is prepared in methanol in a 10mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	GRO-MO Mix	Q-4208	1	1000

The LCS/LCSD must be prepared in the appropriate matrix (organic-free reagent water, or purified solid) depending upon the matrix within the analytical batch; and contain all of the method target analytes. A subset of the method target analytes could be used based on the project specific requirements.

7.4.2.1 For soil/water autosamplers (5mL), a dilution of 10X is required. The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is at or below -10° C.

STATE NOTE: South Carolina and the USACE require that all target analytes are present and evaluated in the LCS.

7.5 Surrogate standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened.

7.5.1 The following are ESC designated volatiles' analysis surrogates:

- toluene-d8
- 4-bromofluorobenzene
- Dibromofluoromethane
- $\alpha\alpha\alpha$ -Trifluorotoluene

Surrogate spiking solutions are purchased from NSI as custom standard Q-4185, or equivalent, at 20,000ug/mL, which contains both internal standards and

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surrogate compounds. This solution is then diluted by 100X to obtain a 200ug/mL working solution.

7.6 Internal standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened. [Internal standard and surrogate standard – NSI custom MS/IS/SS mix Q-4185, or equivalent]

7.6.1 The following are ESC designated volatiles' analysis internal standards:

- 1,4-Difluorobenzene
- 2-Bromo-1chloropropane
- 1,4-dichlorobenzene-d4
- pentafluorobenzene

7.7 4-Bromofluorobenzene (BFB) standard - The BFB in the custom internal standard mix is used to verify mass spectrometer tuning. Since internal standards and surrogates are added to all samples and standards, BFB is included as part of our initial calibration and calibration verification standards. Certificates of analysis must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened.

7.8 Matrix spike (MS) standard - Stock standards must be stored at or below -10° C and have a six month holding time, once opened. Once diluted, the standard must be replaced weekly.

7.8.1 The matrix spike standard is prepared from the stock standard in Section 7.4.2.

7.8.2 The spike should be at a mid-level of the calibration range. Some contracts may require a site-specific concentration.

7.8.3 Standard spiking practice requires the use of ALL TARGET ANALYTES as specified in Table 11.7 and must be evaluated against the current control limits.

Project Specific Requirements (Non-South Carolina Projects): Individual projects may specify required spike compounds. In addition to any project specific requirements, the following table lists the minimum required compounds that must be included in the spike solution.

Minimum Spiking Compounds for Project Specific Requirements	
•	1,1-Dichloroethene
•	Trichloroethene
•	Chlorobenzene
•	Toluene

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Minimum Spiking Compounds for Project Specific Requirements
• Benzene
• n-Hexane (when requested as a target analyte)

All compounds in the spike solution must be evaluated for acceptable recovery. In the absence of established control limits, default recovery limits are 70 - 130%.

7.8.4 STATE NOTE: OHIO VAP SPIKES: Matrix spikes are evaluated using in house limits for only those compounds listed in 7.8.3.

8.0 PROCEDURE

Analysis Summary: Volatile compounds are introduced into the gas chromatograph by purge and trap, via the Archon autosampler, as described on Section 2. If soil samples are high in contamination, a methanolic extraction, SOP No.330760, may be necessary prior to purge and trap analysis. Soils require method 5035 for sample preparation, See SOP 330751.

8.1 Chromatographic conditions: All changes in analytical conditions are listed in the Maintenance Log.

8.1.1 Typical conditions for each instrument and column are listed below:

Inlet	off
Detector	200°C
Oven Equib. Time:	0.50 minutes
Oven Max	240°C
Init Temp	45°C hold 1.0 minute
Ramp	20°C/min to 240 hold 1.0 minute

8.1.2 Typical conditions for each autosampler are listed below:

Heating sample	1 minute at 40°C
Purge	11 minutes at 40°C
Desorb	1 minutes at 250°C
Bake	2 minutes at 260°C

8.1.3 Typical condition for each MS detector are listed below:

Electron energy – 70 volts (nominal)
Mass range – 35 to 300 amu
Scan time – 1.2 sec/scan
Manifold vacuum – 3 x 10⁻⁶ torr

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8.2 **Initial calibration**

- 8.2.1 **TUNING** - Each GC/MS system must be hardware-tuned ($1\mu\text{L} = 50\text{ng}$) with BFB to meet the criteria listed below. The mass-spectrometer must meet acceptable BFB sensitivity criteria before analysis can begin. The instrument must be tuned every 12 hours.

BFB Key Ions and Ion Abundance Criteria	
Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

8.2.2 **Calibration Curve – General Criteria**

- A minimum of 5-point calibration is performed using the primary standards listed in Section 7.4.1. Additional levels may be included to better meet project or client requirements. Regardless of the specific number, the calibration levels analyzed should correspond to a range of concentrations expected to be found in samples, without exceeding the working range of the GC/MS system.
- A calibration point must be analyzed at or below the reporting limit. The concentration of the lowest calibration standard analyzed should be at least 3-5 times the MDL. The instrument response must be distinguishable from the instrument background noise. The signal to noise ratio is the magnitude of the signal strength detected by the mass spectrometer relative to the magnitude of the background noise of the instrument. Instrument conditions must be optimized before the analysis of a calibration curve to minimize background effects.

STATE NOTE: A reporting level standard must be run after calibration is complete. This standard is required by the state of North Carolina and is used to verify the low end of the calibration curve.

STATE NOTE: When analyzing samples from MN, the reporting limit must be verified with each calibration or at least monthly. Verification can be performed by re-quantitation of the

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low calibration standards using the newly updated calibration curve or by analyzing a separate reporting level standard following calibration curve update. This standard must recover +40% of the expected concentration. If the criteria is not met, a higher level standard may be re-quantitated or analyzed; however the reporting limit must be amended to reflect the increased concentration of the standard utilized. Analytes known to be poor performers are dealt with on a case-by-case basis.

- The highest standard must not exceed the linear range of the detector. The concentration of the highest standard must produce a response, which does not cause the MS detector to become saturated. The highest concentration used in the calibration curve must allow the analyte to meet the calibration requirements outlined in Sections 8.2.6.1, 8.2.6.2 and 8.2.6.3.
- When using Method 5035, SOP #330751, the calibration curve must be prepared in the same solutions used to preserve the field samples.
- The method reference spectra must be updated from the mid-point of each calibration.

STATE NOTE: For Ohio VAP samples, when soil samples are received pre-preserved with sodium bisulfate, calibration standards must also include sodium bisulfate at the same concentration. It is preferred that samples are not chemically preserved in the field and are instead capped and shipped to the laboratory to be frozen with 48 hours of sampling.

8.2.3 Calibration Levels for single analytes

- 8.2.3.1 Soil Samples - Soil samples are analyzed with a heated purge in the soil chamber of the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of ICV Mix (See Section 7.4.1) into 5mL of water containing 1g sodium bisulfate.

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Calibration Curve - GC/MS Soil (into 5mL water)	
Intermediate solution volume (μ L)	Concentration of standard (ppb)
0.05	0.5
0.1	1
0.2	2
0.5	5
1	10
2.5	25
5.0	50
10.	100
20.	200

Note 1: When analyzing soil samples by the low-concentration method (Section 8.7.1), the calibration standards must be heated to $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$ prior to purging.

Note 2: Injections should be performed from the lowest to the highest standards with a cleanup injected after the highest standard and followed by the secondary source standard to verify the initial calibration curve.

- 8.2.3.2 Water Samples - Water samples are run with a heated purge using the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of ICV Mix (See Section 7.4.1.) into 5mL of water.

Calibration Curve - GC/MS Water (into 5mL water)	
Intermediate solution volume (μ L)	Concentration of standard (ppb)
0.05	0.5
0.1	1
0.2	2
0.5	5
1.0	10
2.5	25
5.0	50
10.	100
20.	200

- 8.2.3.3 The Centurion autosampler, or equivalent, transfers 5mL of sample to the concentrator for purging and requires full VOA vials. The sampler

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requires 50mL of standard prepared and analyzed using the ICV Mix in Section 7.4.1.

Calibration Curve - GC/MS Water (into 50mL water)	
Intermediate solution volume (μ L)	Concentration of standard (ppb)
0.5	0.5
1	1
2	2
5	5
10	10
25	25
50	50
100	100
200	200

8.2.4 Calibration Levels for GRO by MS

8.2.4.1 Soil Samples - Soil samples are analyzed with a heated purge in the soil chamber of the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of ICV Mix (See Section 7.4.1.) into 5mL of water containing 1g sodium bisulfate.

Calibration Curve - GC/MS Soil (into 5mL water)	
Intermediate solution volume (μ L)	Concentration of standard (ppm)
2	0.4
5	1
10	2
20	4
25	5
35	7
50	10
100	20

8.2.5 Internal Standards and Surrogates – Soil/Water

The autosampler adds 1 μ L of the IS/surrogate mix to each sample. The addition of 1 μ L of the surrogate spiking/internal standard solution to 5mL of sample is equivalent of 40 μ g/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix. Internal Standards are listed Section 7.6.1 and Surrogates are listed in Section 7.5.1.

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Tabulation of the Internal Standards

Tabulate the area response of the characteristic ions (see Attachment III) against each compound's concentration and each internal standard concentration. Then calculate the response factor (RF) for the quantifying ion of each compound relative to the appropriate internal standard according to the calculation provided in Section 9.1. The internal standards used should permit most of the compounds of interest in a chromatogram to have retention times of 0.80 to 1.20, relative to one of the internal standards. The average RF must be calculated and recorded for each compound.

8.2.5 System Performance Check Compounds (SPCCs) – Soil/Water

A system performance check must be made before the calibration curve can be used. The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichlorethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

These compounds are typically used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

Compound	Effect on stability
Chloromethane	This compound is the most likely compound to be lost if the purge flow is too fast.
Bromoform	This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
1,1,2,2-Tetrachloroethane and 1,1-Dichloroethane	Contaminated transfer lines degrade these compounds in purge-and-trap systems. Active sites in trapping materials also can cause problems.

Adjust the purge gas (helium) flow rate to 25-40mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane

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and bromoform. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

8.2.6 Response Factors (RF's) & Calibration Check Compounds (CCC's) - Soil/Water

Using the RF's for the initial calibration curve from Section 8.2.4, calculate and record the percent relative standard deviation (%RSD) for all compounds. Calculate the percent RSD as in Section 9.2. Linearity can be assumed if the RSD criteria is met, thus allowing quantitation calculations to be performed using RF.

8.2.6.1 CCC Criteria - The %RSD for each individual CCC must be less than 30%. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

8.2.6.2 Target Analytes and other Non-CCC's - The RSD must meet the following criteria -

<15% RSD for all 8260B Target Analytes
<35% RSD for all 624 Target Analytes
<20% RSD for all KSGRO Samples
<15% RSD for n-Hexane
<10% RSD for all 601/602 Target Analytes

Compounds not meeting the RSD requirement may be considered for linear regression as stated in 8.2.6.3

8.2.6.3 Linear Regression - Criteria

When any compound does not meet the calibration criteria for RF, the analyst must use linear regression. If linear regression is used, it must be noted on the data (preferably on the CCV RF report), next to the affected compound. It must also meet correlation coefficient criteria of 0.99 or better. USACE requires correlation coefficient criteria of 0.995 or better.

Linear regression is achieved by plotting the instrument response versus the concentration of the standards. The resulting regression line must not be forced through the origin and the origin must not be included as a calibration point.

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Linear regression may not be used for compounds that exceed 40% RSD except for the poor performers listed in section 8.3.4. The RSD for the poor performers cannot exceed a %RSD of 50%. Compound calibration with a %RSD >40 is considered out of control and must be re-calibrated or not used.

For linear regression calculations, see section 9.11.

8.2.6.4 Quadratic Regression - Criteria

In rare instances, quadratic curve fitting may be used; however this type of curve modeling can never be utilized unless historical performance of analytes have exhibited a nonlinear range. Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance.

If a quadratic fit is used, it must be noted on the data (preferably on the CCV RF report), next to the affected compound. It must also meet correlation coefficient criteria of 0.99 or better. USACE requires correlation coefficient criteria of 0.995 or better.

Quadratic regression is achieved by plotting the instrument response versus the concentration of the standards. The resulting regression line must not be forced through the origin and the origin must not be included as a calibration point.

Quadratic modeling may not be used for compounds that exceed 40% RSD except for the poor performers listed in section 8.3.4. The RSD for the poor performers cannot exceed a %RSD of 50%. Compound calibration with a %RSD >40 is considered out of control and must be re-calibrated or not used.

For quadratic model calculations, see section 9.11.

8.2.6.5 Calibration Corrective Action

When the RSD exceeds 15% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

If calibration criteria are not met, then one of the following options must be applied to the GC/MS initial calibration:

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- 8.2.6.5.1 Adjust the instrument and/or perform instrument maintenance and re-analyze the calibration standards until the RSD of the calibration meets criteria.
- 8.2.6.5.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Recalculate the RSD without the low standard to determine if the RSD meets the QC limit. If the lowest standard is dropped, the reporting limit could require a change. Check with the supervisor to determine if a point can be removed and not affect reporting limits requirements.

Compounds that are very soluble in water generally are poor purgers. The ketones, vinyl acetate, acrolein, and acrylonitrile fall into this category.

8.3 Calibration Verification

8.3.1 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) must be analyzed to verify the calibration. This standard must be made from a second source, preferable from a different vendor than the calibration standards. The second source calibration standard must perform within following criteria:

CCC and SPCC compounds	+/- 30%
Other compounds (non poor performers)	+/- 40%
Poor Performers (8.3.2)	in-house LCS limits

8.3.2 CCC's

The curve must be verified initially by a calibration standard known as the Initial Calibration Verification standard (ICV). In addition, the same standard is analyzed every 12 hours for continuing calibration verification (CCV). This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for CCC analytes (Listed in Section 8.2.6.1) and n-Hexane when requested as a target analyte.

Compounds on average response factor uses % difference,

$$\text{% Difference} = (\text{RF}_v - \text{Rf}_{ave}) / \text{Rf}_{ave} \times 100\%$$

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Compounds on regression fit model uses percent drift,

$$\% \text{ Drift} = (\text{Calculated conc} - \text{Theoretic conc}) / \text{Theoretic conc} \times 100\%$$

Criteria for both is <= 20%.

8.3.3 SPCC's

The SPCC's must have a minimum response factor as stated in Section 8.2.5. If these criteria are exceeded, then corrective action is required.

8.3.4 All Target Analytes and Non-CCC's

When analyzing 8260 and 624 concurrently, target analytes and non-CCC compounds must meet the criteria established in 624 (Attachment VI) for all analytes on the 624 list. For analytes not on the 624 list, all target analytes (except for the poor performers, as listed below) and non-CCC's must meet a maximum of 40% drift from the calibration curve. The analyst evaluates all analytes carefully and the experience of the analyst weighs heavily when determining the usability of the data.

Poor performers are allowed a maximum of 50% drift from the calibration curve. The poor performers are as follows:

Poor Performing Compounds

Dichlorofluoromethane
Bromomethane
Chloroethane.
2,2-Dichloropropane.
1,2-Dibromo-3-chloropropane
2-Chloroethylvinyl Ether
Acrolein
Vinyl acetate
Trans-1, 4-dichloro-2-butene
Alcohols (Ethanol, TBA, TAA, ETBA, TBF, Butanol)
Iodomethane.
Naphthalene
1- Methylnaphthalene
2- Methylnaphthalene
Acetone
2-Butanone
2-Hexanone
4-Methyl-2-pentanone

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8.3.5 Laboratory Control Standard (LCS)

- Must be prepared near the mid-point of the calibration curve for each batch of samples.
- QC Limits for the laboratory control standard are in Table 11.7, LCS and MS/MSD Criteria.
- If the stated criteria are exceeded, then corrective action is required.
- See Section 11.7.1, on marginal exceedences.
- **STATE NOTE:** All **South Carolina** LCS responses must be within 70 – 130%. NO FAILURES are acceptable, qualifiers cannot be used. FAILURES require a batch re-analysis. See Section 10 for QC evaluation.

8.3.6 Internal Standard Evaluation

8.3.6.1 When a calibration is performed at the beginning of an analytical run: The internal standard areas must be evaluated against the mid-point of the curve. Samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against mid-point of the curve. Then a CCV is analyzed, this is compared to the mid-point of the initial calibration curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

8.3.6.2 When an analytical run is started using a passing ICV (which is compared against the initial calibration mid-point to verify the calibration curve): Samples are analyzed within a 12-hour window, the internal standards of those samples are evaluated against the daily ICV. Then a CCV is analyzed, this is compared to the mid-point of the curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

8.4 Gas chromatographic analysis:

8.4.1 Typical sequence order for loading the autosampler with calibration:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Calibration standard(s)	Initial volatiles calibration and 7-point for GRO (if analyzed)
Second Source Calibration	Verify initial calibration with second source.

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Sample/QC Type	Use
Verification (SSCV)	
Laboratory Control Sample/ Laboratory Control Sample Duplicate	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

8.4.2 Typical sequence order for loading the autosampler with no calibration:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Initial Calibration Verification (ICV)	Verify initial calibration.
Laboratory Control Sample/ Laboratory Control Sample Duplicate	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

8.5 GC/MS Analysis -- Water Samples

- 8.5.1 Screening the sample prior to purge-and-trap analysis provides guidance on whether sample dilution is necessary and prevents contamination of the purge-and-trap system. Three screening techniques that can be used are the headspace sampler, using a gas chromatograph (GC) equipped with a photo ionization detector (PID) in series with an electrolytic conductivity detector

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(ELCD) (SW-846 Method 3810), extraction of the sample with hexadecane and analysis of the extract on a GC with a FID and/or an ECD (SW-846 Method 3820), and screening of 5mLs of sample using an HNU or equivalent portable PID.

- 8.5.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.
- 8.5.3 Set up the GC/MS system as outlined in Section 8.1.
- 8.5.4 BFB tuning criteria and GC/MS calibration criteria must be met (Section 8.2.1) before analyzing samples.
- 8.5.5 Rinse a 5mL syringe by filling and discarding 3 times with distilled water. Open the sample and quickly remove a 5mL aliquot. Immediately replace the sample cap and deposit the aliquot into a clean/baked 40mL autosampler vial. Cap quickly and tightly.

All samples analyzed for USACE must be run using the autosampler prep technique. VOA vials for USACE samples must not be opened prior to analysis to minimize target analyte loss.

After the sample has been loaded on the instrument, check the pH of the sample using the remaining sample in the VOA vial. Use universal pH paper and record the sample pH to the nearest whole pH unit. Samples not passing the pH requirements are flagged with a "T2" qualifier.

- 8.5.6 Sample Dilution -- When necessary, samples can be diluted before purging. This can be performed in a clean/baked 40mL vial that has been filled with 5mL of measured reagent water. The sample is measured through the use of an appropriate microliter syringes.
- 8.5.7 Compositing samples prior to GC/MS analysis – Site or project-specific requirements may require compositing of samples, which is performed according to the instructions below. Compositing of samples is only performed at the request of the client.
 - 8.5.7.1 Add 5mL of each sample (up to 5 samples are allowed) to a 25mL glass syringe. Practice special precautions to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be composited, as long as equal volumes of each sample are used.
 - 8.5.7.2 The samples must be cooled to 4°C or less during composition to minimize the loss of volatiles. Sample vials may be placed in a tray of ice to prevent volatile loss during this process.

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8.5.7.3 Mix each vial well. Using the 25mL syringe, draw out a 5mL aliquot of sample.

8.5.7.4 Once all the aliquots have been combined in the syringe, invert the syringe several times to mix the aliquots. The sample is now ready to be analyzed.

8.5.7.5 If less than five samples are being used for compositing, a smaller syringe may be used, provided ample volume is obtained for analysis.

NOTE: Samples are not be routinely composited, however, if site-specific requirements state procedures for compositing samples, the laboratory makes every effort to comply with those requirements.

8.5.8 Surrogate/Internal Standards – The autosampler adds 1uL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution to 5mL of sample is equivalent of 40µg/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix.

8.5.9 Refer to SOP 330752 EPA 5030B Purge and Trap for Aqueous Samples and SOP 330751 EPA 5035 for additional information on sample introduction.

8.5.10 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Secondary ion quantitation is allowed only when there are sample interferences with the routinely quantitated primary ion. When a sample is analyzed that has saturated the detector, the samples following must be analyzed for contamination. If any sample shows contamination, they must be re-analyzed.

8.6 GC/MS Analysis -- Water-miscible liquids

8.6.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with laboratory water.

8.6.2 Initial and serial dilutions can be prepared by pipetting a known amount of the sample to a 50mL volumetric flask and diluting to volume with organic-free reagent water. Transfer immediately to a clean/baked 40mL vial using a 5mL syringe.

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- 8.6.3 Alternatively, prepare dilutions directly in a clean/baked 40mL vial filled with organic-free reagent water by adding at least 1µL, but not more than 2500µL of liquid sample. The sample is ready for addition of internal and surrogate standards. Proceed with Section 8.5.8.

8.7 GC/MS Analysis -- Sediment/soil and waste samples

These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. The screening of samples is highly recommended. Screening data should be used in conjunction with site-specific DQOs, if known, to determine whether to use the low-concentration method (0.005 - 1 mg/Kg) or the high-concentration method (> 1 mg/Kg).

- 8.7.1 Low-concentration method -- This is designed for samples containing individual purgeable compounds of <1 mg/Kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards. All QC samples and standards are to be analyzed under the same conditions as the samples, using 5g of clean sand or clean soil.

STATE NOTE: This option cannot be used for OH VAP or South Carolina samples. Please refer to SOP 330751 that addresses Method 5035 for sample preparation.

- 8.7.1.1 Use a 5g sample if the expected concentration is <0.1mg/Kg, or a 1g sample for expected concentrations between 0.1 and 1mg/Kg.

- 8.7.1.2 The GC/MS system must be set up as in Sections 8.1 and 8.2. This must be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. Both the initial and daily calibration standards (Sections 8.2 and 8.3) must be heated to 40°C purge temperature. Refer to Method 5035 (SOP #330751) for additional instructions for 8260B soil analysis.

- 8.7.1.3 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. Weigh the amount determined in Section 8.7.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

- 8.7.1.4 Add distilled water to the purging vial, which contains the weighed amount of sample, and place the vial in the purge-and-trap system.

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NOTE: Prior to the placement of the vial, the procedures in Sections 8.7.1.4 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

8.7.2 High-concentration method -- The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e., petroleum and coke wastes) that are soluble in methanol are diluted. An aliquot of the extract is added to organic-free reagent water containing surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of >1.0 mg/Kg must be analyzed by this method. See SOP #330753 *Waste Dilution for VOC's* for additional information.

STATE NOTE: This method is not suitable for samples from South Carolina. South Carolina does not recognize the practices in sections 8.7.2.1 or 8.7.2.2. 5035 must be used for all high-level soil samples, see SOP 330751.

8.7.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol, weigh 5g (wet weight) of sample into a tared 40-mL vial. Use a top-loading balance. Note and record the actual weight to 0.1g. For waste that is soluble in methanol, tetraglyme, or PEG, weigh 5g (wet weight) into a 40mL vial.

8.7.2.2 Add 5mL Methanol. Shake well for 2 minutes. See SOP 330751.

NOTE: Sections 8.7.2.1 and 8.7.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

8.7.2.3 The GC/MS system must be set up as in Sections 8.1.

8.7.2.4 If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start with 100µL.

8.7.2.5 In a clean/baked vial filled with reagent water, inject the corresponding aliquot of methanol extract. Immediately cap and place in the autosampler. The autosampler adds 1uL of the IS/surrogate mix to all of the samples.

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- 8.7.2.6 Proceed with the analysis as outlined in Sections 8.5.9-8.5.10. Analyze all blanks on the same instrument as that used for the samples. The standards and blanks must also contain 100µL of the dilution solvent to simulate the sample conditions.
- 8.7.2.7 For a matrix spike in the high-concentration of sediment/soil samples, Add a 100µL aliquot of this extract to 5mL of organic-free reagent water for purging (as per Section 8.7.2.6) in a clean/baked 40mL VOA vial and add 20µL spiking solution, 1µL internal and surrogate standard solution (IS/Surr solution added by autosampler).

8.8 Qualitative Identification

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. All hits must be visually compared to the reference spectrum for confirmation. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the criteria below are met.

- 8.8.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- 8.8.2 The RRT of the sample component is within + 0.06 RRT units of the RRT of the standard component.
- 8.8.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum the corresponding abundance in a sample spectrum can range between 20% and 80%).
- 8.8.4 Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

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8.8.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound.

8.8.6 TIC's - Tentatively Identified Compounds

Periodically, clients may request additional identification of compounds that are not normally calibrated. This identification is limited to the compounds in the current mass spectral library employed by ESC.

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification is determined by the type of analyses being conducted. At the client request, when serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) must always be reported. Guidelines for making tentative identification are:

- (1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within 30% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum the corresponding sample ion abundance must be between 20 and 80%).
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

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Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches does the mass spectral interpretation specialist assign a tentative identification.

8.9 Quantitative Analysis

- 8.9.1 When a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is accomplished using the internal standard technique, as described in Section 9. The internal standard used must be the one nearest the retention time of that of a given analyte.
- 8.9.2 Sediment/soil samples are reported on a dry weight basis, while sludge and wastes are reported on a wet weight basis. The percent dry weight of the sample (see Section 9.7) must be reported along with the data in either instance. At ESC, the dry weight conversion calculations for sample reporting are performed by the LIMS system. [Dry weight only when requested]. The LIMS Final Client Report represents the reporting basis as either wet weight or dry weight, depending upon the calculation used.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Response Factor (RF)

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate, in $\mu\text{g/L}$.

C_{is} = Concentration of the internal standard, in $\mu\text{g/L}$.

9.2 Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100\%$$

where:

RSD = Relative standard deviation.

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RF = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

9.3 Percent Difference

$$\frac{RF_v - RF}{RF} * 100\%$$

where:

RF = Average response factor from initial calibration.
RF_v = Response factor from current verification check standard.

9.4 Percent Drift

$$\% \text{Difference} = \frac{C_0 - C_1}{C_1} \times 100$$

where:

C₁ = compound standard concentration
C₀ = measured concentration using selected quantitation method

9.5 Concentration of Target Analytes in Water and Water-Miscible Waste

$$\text{Concentration(ug/L)} = \frac{(A_x)(I_s)(D)}{(A_{is})(\text{ave.RF})(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
I_s = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
ave.RF = Mean response factor from the initial calibration.
V_s = Volume of the aqueous sample extracted or purged (mL). If units of liters are used for this term, multiply the results by 1000.

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9.6 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

9.6.1 High-concentration procedure

$$\text{Concentration(ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:

A_x, I_s, A_{is}, ave.RF are the same as in water and water-miscible waste above.

V_t = Volume of total extract (μL) (use 10,0000 μL or a factor of this when dilutions are made).

V_i = Volume of extract added (μL) for purging.

W_s = Weight of sample extracted or urged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

9.6.2 Low-concentration procedure

$$\text{Concentration(ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:

A, I_s, A_{is}, RF are the same as in water and water-miscible waste above.

V_t = Volume of total extract (μL) (use 10,0000 μL or a factor of this when dilutions are made).

V_i = Volume of extract added (μL) for purging.

W_s = Weight of sample extracted or urged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

9.6.2 Soil Weight determination with Methanol (samples received with MeOH).

$$\text{SoilWeight} = \text{VialTotalWeight(vial,soil,MeOH)} - \text{TareVialWeight} - \text{MeOHWeight}.$$

9.7 Percent Dry Weight

$$\% \text{DryWeight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

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9.8 Percent Recovery (%R)

$$\% R = \frac{\text{Measured concentration}}{\text{Actual concentration}} \times 100$$

9.9 Relative Percent Difference (RPD)

$$RPD = \frac{| \text{Sample dup1} - \text{Sample dup2} |}{\{(\text{Sample dup1} + \text{Sample dup2}) / 2\}} \times 100$$

where:

Sample dup1 = the measured concentration of the first sample duplicate
Sample dup2 = the measured concentration of the second sample duplicate

9.10 Correlation Coefficient and Regression Equations

The correlation coefficient is the measure of the degree to which x (the instrument response) and y (standard concentration) relate to each other as a change occurs in one. The correlation coefficient (r) is expressed as:

$$r = \frac{\sum d_x d_y}{\sqrt{(\sum d_x^2)(\sum d_y^2)}}$$

where: d_x and d_y are the deviations from each mean.

9.11 Linear Regression and Quadratic Regression – Forcing through the origin is NOT allowed.

STATE NOTE: For Ohio VAP samples, quadratic curve fitting is only allowed for analytes that do not historically exhibit a linear response. This calibration model may not be utilized to extend the calibration range or bypass instrument maintenance.

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For a first order linear regression for:

$$Y = a + bX$$

$$b = \frac{\sum xy - nXY}{\sum x^2 - nX^2}$$

where:

x – the instrument response
y - standard concentration
X - the average instrument response
Y- the average concentration
n – the number of standards

Quadratic regression is mathematically defined as:

$$y = ax^2 + bx + c$$

Quadratic regression is utilized to find a parabola of “best fit” for a set of data generated from the instrument response and the standard concentration.

- 9.12 For required method performance criteria, see section 10 and 11. For corrective actions, see section 11.0.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

- 10.2 Batches:

10.3.1 Extraction Batches:

Extraction batches are defined as sets of 1 - 20 samples. Extraction batches must include the following: 1 method blank, 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair (if sufficient sample is available). Exceptions are made for waste dilution samples where the minimum batch QC must include a blank, an LCS/LCSD pair.

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Additional instructions on Batch QC including required frequency, acceptance criteria and corrective actions can be found in Section 11 and Tables 11.1 & 11.7.

10.3.2 Analytical Batches (sequences):

Analytical batches analysis must include the following: 1 Initial Calibration Verification (ICV) and BFB tune at the beginning of run, 1 Continuing Calibration Verification (CCV) and BFB tune every 12 hours.

- 10.3 Perform BFB tune every 12 hours for 8260B, and 6200B. BFB tuning for method 624 is every 24 hours. Tuning acceptance criteria are presented in Section 8.2.1. The computer software automatically evaluates the tune information. The analyst must be aware of the process used. The criteria specified in Section 8.2.1 would typically be derived using three scans: one at the apex and one on both sides of the apex.
- 10.4 Run a minimum of a 5-point initial calibration curve (3-point can be used if 624/6200B are being run independently of 8260B), using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria for initial calibration are presented in Section 8.2. Calibration is verified by analyzing Second Source Calibration Verification (SSCV) standard; acceptance criteria for the SSCV is presented in Section 8.3.1.
- 10.5 Run a mid-point Initial Calibration Verification (ICV) using the primary source standards every 12 hours before sample analysis. Also run a CCV every 12 hours during an analytical sequence for 8260B, 624 and 6200B. See sections 8.3.2 – 8.3.6 for acceptance criteria.
- 10.6 Retention Time Windows:
 - 10.6.1 To determine retention time windows for each component in the calibration mix, make three injections of the mid-level standard over the course of 72 hours. Calculate the standard deviation of the three retention times for each analyte in the mix. The retention time window is defined as the mean retention time plus or minus three times the standard deviation of the retention times established during the 72-hour period for each component. The typical estimated retention time windows are set at +/- 0.05 minutes. However, the experience of the analyst weighs heavily in the interpretation of chromatograms. If the standard deviation for a component is zero, substitute the standard deviation of a closely eluting compound to develop a valid retention time window.
 - 10.6.2 Retention time windows must be re-calculated whenever a new column is installed or whenever a major modification has been made to the instrument system.

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- 10.6.3 Compounds are identified if they fall within the retention time window specified for that compound.
 - 10.6.4 If the retention time for any internal standard changes by more than 30 seconds from the mid-point of the current initial calibration curve, the chromatographic system must be inspected for malfunction and corrections must be made, as required.
 - 10.7 Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, polishing detector windows, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.
- 11.0 DATA VALIDATION AND CORRECTIVE ACTION
- 11.1 SITE-SPECIFIC requirements and STATE SPECIFIC criteria must be reviewed and used, if known, for data review.

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. blank, surrogate, spike, ISTD, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.
 - 11.2 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the MDL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.
 - 11.4 INITIAL AND CONTINUING CALIBRATION VERIFICATION STANDARD: An Initial Calibration Verification (ICV) standard is analyzed before sample analysis can begin. Ensure that a continuing calibration verification (CCV) standard was analyzed every 12 hours and meets the criteria in Section 8.3. If these criteria are exceeded, corrective action is necessary. If the source of the problem cannot be determined after corrective action has been taken, a new calibration must be generated. This criterion must be met before sample analysis begins and/or re-analysis of all samples up to the last acceptable CCV standard.

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- 11.5 METHOD BLANK ASSESSMENT - The analyst must confirm that this blank was analyzed at the required frequency of 1 per batch of 20 samples.

The method blank must not exhibit any contamination of any analyte above ½ the report limit for any of the method target analytes. Corrective action must be performed any time method target analytes are detected above the ½ the report limit to reduce and control contamination.

Corrective Action/ Evaluation:

Blank contamination above ½ the reporting limit - Assess the effect on the samples. Samples containing detectable concentrations at or just above the reporting limit may require re-analysis. Samples with concentrations below the reporting limit require no corrective action.

Blank contamination above the report limit – All samples containing detectable amounts above the reporting limit must be re-analyzed or qualified. Samples with no detectable amounts above the reporting limit do not require re-analysis, but the samples must be qualified with blank contamination and it must be mentioned in the case narrative in the data package.

Instrument blanks may be injected at any time in the sequence to verify absence of contamination. The source of contamination must be investigated and reduced or eliminated. Any time contamination is noted in the method blank, the situation and impact on the data should be discussed in the case narrative.

- 11.6 LABORATORY CONTROL SAMPLES - Assess that LCSs were prepared at the required frequency of 1 per batch of 20. If the recovery does not meet criteria, see section 11.7.1 for marginal failures. If it is still out of control limits, then all field and QC samples in the batch must be re-analyzed.

REQUIRED RE-ANALYSIS - None of the following compounds can recover beyond established criteria: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. If any of these compounds fail, the LCS/LCSD and all affected batch samples must be re-analyzed.

STATE NOTE: OHIO VAP LCS/LCSD: LCS's are evaluated only for the compounds listed in 7.8.3. The LCS/LCSD contain all method target analytes. For OHIO VAP and project specific requirements, the LCS/LCSD contains, at a minimum: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, benzene, and n-hexane (Ohio VAP) and must meet both the acceptance criteria for accuracy and precision found in Table 11.7.

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STATE NOTE: SOUTH CAROLINA LCS: responses must be within 70 – 130%. No failures are acceptable; Qualifiers cannot be used. Failures require a batch re-analysis.

Routine LCS Control limits are in Table 11.7. Qualifiers must be applied to any LCS compound that does not meet these criteria and are considered out of control. The percent difference for all method target analytes must be within QC RPD limits. If not, re-analyze the duplicate(s) or prepare a new calibration curve, as necessary.

11.7 LCS/LCSD & MS/MSD CRITERIA

**Table 11.7 Laboratory Control Standard and Matrix Spike QC Limits
(effective 10/21/04)**

**ESC Water Acceptance Criteria for LCS and MS/MSD
(Limits are subject to change – State Specific Limits may apply*)**

ANALYTE	LCS/LCSD LIMITS *	RPD LIMITS	MS/MSD LIMITS*	RPD LIMITS
Dichlorodifluoromethane	42 - 161	26	41 - 139	22
Chloromethane	57 - 139	17	45 - 150	16
Vinyl Chloride	60 - 131	12	40 - 143	20
Bromomethane	24 - 117	17	34 - 110	34
Chloroethane	49 - 143	22	43 - 156	17
Trichlorofluoromethane	60 - 130	12	53 - 123	18
Ethyl Ether	63 - 150	17	61 - 143	18
Acrolein	28 - 115	29	21 - 105	12
1,1-Dichloroethene	67 - 137	10	55 - 143	17
1,1,2-Trichloro-1,2,2-trifluoroethane	73 - 130	10	63 - 139	17
Acetone	36 - 156	38	20 - 148	19
Iodomethane	54 - 118	12	50 - 109	19
Carbon Disulfide	56 - 161	11	50 - 160	17
Methylene Chloride	60 - 125	10	59 - 118	16
Acrylonitrile	47 - 119	18	44 - 118	17
trans-1,2-Dichloroethene	64 - 138	9	63 - 124	17
Methyl Tert Butyl Ether	63 - 126	11	59 - 126	18
1,1-Dichloroethane	50 - 140	11	60 - 117	15
Vinyl Acetate	57 - 133	20	52 - 130	16
Di Isopropyl Ether	55 - 135	11	59 - 120	15
2,2-Dichloropropane	64 - 128	10	60 - 119	16

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ANALYTE	LCS/LCSD LIMITS *	RPD LIMITS	MS/MSD LIMITS*	RPD LIMITS
cis-1,2-Dichloroethene	64 - 124	12	67 - 112	16
2-Butanone (MEK)	46 - 127	24	41 - 124	23
Bromochloromethane	66 - 128	9	73 - 123	15
Tetrahydrofuran	40 - 126	20	42 - 111	23
Chloroform	67 - 119	10	66 - 111	13
1,1,1-Trichloroethane	52 - 131	12	57 - 118	14
Carbon Tetrachloride	60 - 138	16	63 - 124	13
1,1-Dichloropropene	59 - 127	11	61 - 118	14
Benzene	55 - 129	11	57 - 124	14
1,2-Dichloroethane	56 - 132	12	61 - 127	13
Trichloroethene	68 - 117	11	67 - 109	14
1,2-Dichloropropane	62 - 121	14	64 - 115	16
Dibromomethane	72 - 116	10	72 - 112	14
Bromodichloromethane	68 - 120	9	70 - 114	12
2-Chloroethylvinyl Ether	41 - 115	22	20 - 138	29
cis-1,3-Dichloropropene	68 - 120	17	66 - 110	13
4-Methyl-2-Pentanone (MIBK)	58 - 129	15	57 - 123	17
Toluene	71 - 114	12	68 - 108	14
trans-1,3-Dichloropropene	57 - 104	12	55 - 100	15
1,1,2-Trichloroethane	71 - 117	9	46 - 131	22
Tetrachloroethene	67 - 124	13	62 - 115	13
1,3-Dichloropropane	71 - 114	11	72 - 110	11
2-Hexanone	52 - 133	16	59 - 115	15
Chlorodibromomethane	67 - 117	11	67 - 111	20
1,2-Dibromoethane	71 - 117	13	71 - 114	16
Chlorobenzene	71 - 121	11	70 - 113	13
1,1,1,2-Tetrachloroethane	68 - 125	10	70 - 114	11
Ethylbenzene	70 - 121	10	69 - 109	14
m&p-Xylene	70 - 122	12	67 - 110	15
o-Xylene	71 - 116	11	71 - 111	13
Styrene	71 - 116	12	64 - 112	11
Bromoform	70 - 124	10	64 - 112	11
Isopropylbenzene	71 - 123	12	65 - 122	14
Bromobenzene	73 - 123	13	69 - 113	15
1,1,2,2-Tetrachloroethane	63 - 127	13	68 - 121	13

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ANALYTE	LCS/LCSD LIMITS *	RPD LIMITS	MS/MSD LIMITS*	RPD LIMITS
1,2,3-Trichloropropane	56 - 130	16	66 - 121	11
trans-1,4-Dichloro-2-Butene	52 - 138	13	64 - 114	12
n-Propylbenzene	67 - 124	11	58 - 120	13
2-Chlorotoluene	68 - 121	12	63 - 111	16
4-Chlorotoluene	67 - 126	14	66 - 110	13
1,3,5-Trimethylbenzene	67 - 121	15	67 - 110	15
tert-Butylbenzene	66 - 125	14	65 - 109	13
1,2,4-Trimethylbenzene	64 - 119	14	66 - 114	17
sec-Butylbenzene	67 - 122	11	63 - 108	17
1,3-Dichlorobenzene	67 - 126	13	66 - 112	16
p-Isopropyltoluene	65 - 125	13	64 - 111	14
1,4-Dichlorobenzene	72 - 118	13	64 - 113	17
1,2,3-Trimethylbenzene	63 - 103	12	60 - 111	12
1,2-Dichlorobenzene	74 - 115	11	55 - 98	12
n-Butylbenzene	68 - 124	16	67 - 109	14
1,2-Dibromo-3-Chloropropane	51 - 127	15	54 - 110	14
1,2,4-Trichlorobenzene	71 - 136	18	56 - 109	16
Hexachlorobutadiene	64 - 107	18	52 - 125	17
Naphthalene	77 - 140	16	49 - 104	15
1,2,3-Trichlorobenzene	73 - 143	14	51 - 143	26
SURROGATE LIMITS				
Dibromofluoromethane	79 - 121			
a,a,a-Trifluorotoluene	86 - 113			
Toluene-d8	79 - 118			
4-Bromofluorobenzene	76 - 122			

***STATE NOTE:** SOUTH CAROLINA requires that ALL LCS compounds meet the 70 – 130% acceptance criteria for all target analytes and that all MS/MSD compounds meet the established criteria for all target analytes. No qualifiers are accepted.

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GENERAL ESC Soil Acceptance Criteria for LCS and MS/MSD
(Limits are subject to change – State Specific Limits may apply*)

ANALYTE	LCS/LCSD LIMITS *	RPD LIMITS	MS/MSD LIMITS*	RPD LIMITS
Dichlorodifluoromethane	71 - 152	16	34 - 129	13
Chloromethane	50 - 153	15	56 - 138	12
Vinyl Chloride	58 - 136	13	56 - 120	20
Bromomethane	34 - 129	27	34 - 99	12
Chloroethane	47 - 135	16	45 - 145	23
Trichlorofluoromethane	51 - 132	15	52 - 107	16
Ethyl Ether	76 - 134	12	68 - 144	9
Acrolein	43 - 104	17	25 - 77	13
1,1-Dichloroethene	65 - 129	13	62 - 127	15
1,1,2-Trichloro-1,2,2-trifluoroethane	65 - 142	13	60 - 121	16
Acetone	48 - 155	20	33 - 112	20
Iodomethane	41 - 124	17	41 - 119	20
Carbon Disulfide	60 - 154	19	50 - 156	16
Methylene Chloride	60 - 127	14	46 - 126	12
Acrylonitrile	54 - 123	15	51 - 108	8
trans-1,2-Dichloroethene	67 - 137	16	66 - 119	15
Methyl Tert Butyl Ether	74 - 123	12	58 - 126	13
1,1-Dichloroethane	57 - 133	14	60 - 116	12
Vinyl Acetate	60 - 142	17	27 - 105	33
Di Isopropyl Ether	65 - 123	13	62 - 119	12
2,2-Dichloropropane	59 - 133	15	54 - 114	14
cis-1,2-Dichloroethene	67 - 126	13	63 - 114	13
2-Butanone (MEK)	57 - 133	14	49 - 124	13
Bromochloromethane	73 - 131	13	66 - 133	8
Tetrahydrofuran	52 - 124	29	39 - 142	20
Chloroform	69 - 119	12	50 - 122	13
1,1,1-Trichloroethane	69 - 112	12	59 - 110	16
Carbon Tetrachloride	63 - 133	13	65 - 109	14
1,1-Dichloropropene	69 - 119	15	60 - 108	15
Benzene	72 - 115	13	62 - 113	14
1,2-Dichloroethane	71 - 119	11	60 - 118	9
Trichloroethene	71 - 120	13	62 - 112	15
1,2-Dichloropropane	72 - 119	14	56 - 124	10

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ANALYTE	LCS/LCSD LIMITS *	RPD LIMITS	MS/MSD LIMITS*	RPD LIMITS
Dibromomethane	74 - 123	14	66 - 123	8
Bromodichloromethane	72 - 125	14	66 - 121	10
2-Chloroethylvinyl Ether	34 - 132	16	32 - 126	43
cis-1,3-Dichloropropene	74 - 121	16	63 - 118	12
4-Methyl-2-Pentanone (MIBK)	65 - 137	16	63 - 123	10
Toluene	73 - 117	15	59 - 116	22
trans-1,3-Dichloropropene	63 - 108	14	49 - 103	11
1,1,2-Trichloroethane	77 - 115	14	68 - 117	10
Tetrachloroethene	72 - 123	15	54 - 116	10
1,3-Dichloropropane	77 - 114	14	61 - 124	8
2-Hexanone	69 - 126	16	59 - 116	11
Chlorodibromomethane	74 - 117	15	64 - 114	15
1,2-Dibromoethane	74 - 121	14	68 - 118	10
Chlorobenzene	76 - 119	13	62 - 119	9
1,1,1,2-Tetrachloroethane	76 - 120	13	64 - 126	9
Ethylbenzene	72 - 121	14	64 - 113	11
m&p-Xylene	73 - 121	15	62 - 113	16
o-Xylene	71 - 125	15	65 - 120	16
Styrene	71 - 124	16	59 - 115	10
Bromoform	68 - 137	15	61 - 130	12
Isopropylbenzene	71 - 128	17	65 - 114	11
Bromobenzene	75 - 129	15	54 - 134	9
1,1,2,2-Tetrachloroethane	75 - 122	19	72 - 110	12
1,2,3-Trichloropropane	72 - 122	16	59 - 122	16
trans-1,4-Dichloro-2-Butene	51 - 141	19	56 - 116	15
n-Propylbenzene	68 - 130	18	59 - 108	14
2-Chlorotoluene	72 - 122	18	56 - 111	9
4-Chlorotoluene	72 - 126	18	55 - 108	10
1,3,5-Trimethylbenzene	70 - 124	19	60 - 110	16
tert-Butylbenzene	68 - 130	19	63 - 115	11
1,2,4-Trimethylbenzene	67 - 124	18	56 - 110	18
sec-Butylbenzene	69 - 129	19	60 - 107	12
1,3-Dichlorobenzene	72 - 127	18	45 - 113	12
p-Isopropyltoluene	66 - 135	17	54 - 108	11
1,4-Dichlorobenzene	71 - 121	15	40 - 113	10
1,2,3-Trimethylbenzene	62 - 106	13	47 - 102	22

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ANALYTE	LCS/LCSD LIMITS *	RPD LIMITS	MS/MSD LIMITS*	RPD LIMITS
1,2-Dichlorobenzene	78 - 114	13	48 - 117	12
n-Butylbenzene	69 - 126	13	44 - 97	16
1,2-Dibromo-3-Chloropropane	62 - 127	19	48 - 118	15
1,2,4-Trichlorobenzene	67 - 156	17	41 - 105	16
Hexachlorobutadiene	65 - 113	13	34 - 89	16
Naphthalene	64 - 159	14	54 - 139	17
1,2,3-Trichlorobenzene	68 - 158	15	45 - 114	11

SURROGATE LIMITS

Dibromofluoromethane	74 - 126
a,a,a-Trifluorotoluene	85 - 113
Toluene-d8	87 - 112
4-Bromofluorobenzene	73 - 127

***STATE NOTE:** SOUTH CAROLINA requires that ALL LCS compounds meet the 70 – 130% acceptance criteria for all target analytes and that all MS/MSD compounds meet the established criteria for all target analytes. No qualifiers are accepted.

Control limits for laboratory control samples, matrix spikes and surrogates must be updated semi-annually. Even though control charts must be maintained for the LCS, MS and surrogates, the acceptance criteria derived from these charts may not be appropriate if the charted ranges are too wide. Corrective action should be performed to improve analytical procedures and/or instrument conditions in any case where the charted ranges are too wide and new limits generated.

The RPD of batch LCS/LCSD's should fall within the control limits determined from the precision control charts. However, if the RPD is outside these control limits, the batch will not be rejected, as long as the LCS recovery is acceptable. This precision information should be evaluated to see how systematic problems could be identified. If problems are suspected, the method should be fully evaluated.

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11.7.1 Quality control criteria must be checked for the LCS and LCSD.

LCS or LCSD samples that do not pass the acceptable QC criteria must be re-analyzed. LCS failures can meet marginal exceedence criteria below. Normal compound list for 8260/624 contains typical 90 analytes; therefore only five analyte can be considered as marginal excedences. If the failure persists, re-prepare and re-analyze the entire sample batch.

When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside of control limits. Upper and lower marginal exceedence (ME) limits are established by +/- four times the standard deviation. The number of marginal exceedence is based on the number of analytes in the LCS.

Number of allowable marginal excedences:

90 analytes, 5 analytes allowed in the ME limit
71 – 90 analytes, 4 analytes allowed in the ME limit.
51 – 70 analytes, 3 analytes allowed in the ME limit.
31 – 50 analytes, 2 analytes allowed in the ME limit.
11 – 30 analytes, 1 analyte allowed in the ME limit.
< 11 analytes, no analyte allowed in the ME limit.

Marginal excedences must be random events.

STATE NOTE: For South Carolina and Ohio VAP samples, marginal excedences do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

11.8 MATRIX SPIKE ASSESSMENT: Assess that matrix spike/matrix spike duplicates were analyzed at required frequency of 1 per batch of 20.

- The analyst also verifies that the samples were spiked at the appropriate level.
- The order of preference for spiking levels is as follows:
 - 1) If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two;
 - 2) if an action level exists, spike at this level
 - 3) if neither of the first two conditions apply, spike at a level that corresponds between the low and mid-level calibration standards.
- Acceptance criteria are that all %Recovery and/or RPD results must be within the indicated control limits on the appropriate MS control charts. See Table 11.7 for LCS/LCSD & MS/MSD limits and QC acceptance. If these conditions are not met, perform the following corrective actions as appropriate.
- If both LCS/LCSD and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be re-analyzed.

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- If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Re-analyze to verify the matrix effect. If a matrix effect is still suspected, then the project manager must be contacted to discuss further alternatives and the potential impact on the project. Reported data must be flagged. Reasonable attempts must be made to address matrix interference.

STATE NOTE: South Carolina requires that all target compounds meet the established MS/MSD criteria. No qualifiers can be applied, except in the circumstance where matrix interference is apparent and confirmed.

STATE NOTE: OHIO VAP SPIKES: Matrix spikes are evaluated only for the compounds listed in 7.8.3.

PROJECT SPECIFIC CRITERIA (Non-South Carolina Samples): Acceptance criteria are that all %Recovery and/or RPD results meet project-established goals.

- 11.9 MATRIX SPIKE DUPLICATES: Assess that matrix duplicates were analyzed at required frequency. Acceptance criteria are that all RPD results must be within the indicated control limits on the appropriate precision control charts in Table 11.7. If these conditions are not met, perform the following corrective actions as appropriate.
- Re-analyze the sample to verify a matrix effect.
 - If the duplicate precision is still unacceptable, and LCS precision is acceptable, then a potential matrix effect has been identified. The project manager must be contacted to discuss further alternatives and the potential impact on the project.

PROJECT SPECIFIC CRITERIA (Non-South Carolina Samples): Acceptance criteria are that all RPD results meet project-established goals.

- 11.10 SURROGATE EVALUATION: Check the surrogate calculations for correctness for all samples, blanks, ICV/CCV/SSCV, LCS/LCSD, MS, MSD, and MD. The following acceptance criteria apply to surrogate recoveries: The surrogate recoveries for all QC samples must be within established control limits. If the surrogate recoveries are outside limits for Blank, ICV/CCV/SSCV, and LCS/LCSD, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary. All samples associated with batch or sequence needs to be re-analyzed. The surrogate recoveries for all field samples must be within established control limits. If more than two surrogates recoveries are outside limits, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary which includes qualifying data with J1 (outside upper limit) or J2 (outside lower limit). When one or two surrogates fail, data is qualified with J1 (outside upper limit) or J2 (outside lower limit).

STATE NOTE: OHIO VAP: The surrogate recoveries for all field and QC samples must be within established control limits. If the surrogate recoveries are outside limits, re-

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analysis (undiluted) must be performed for verification. If recoveries are still outside control limits, corrective action is necessary which includes qualifying data with J1 (outside upper limit) or J2 (outside lower limit). The sample(s) needs to be re-analyzed to confirm the failure or following corrective actions.

11.11 INTERNAL STANDARD AREA COUNT: The analyst checks the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area response for any of the internal standards changes by a factor of two (-50% to +100%) as per section 8.3.6, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered.

- Check to be sure there are no errors in the internal standards preparation or addition. Also check instrument performance.
- If any internal standard criteria fails high (> +100%), sample must be re-analyzed with possible dilution. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J9 (IS high, data is likely to show low bias).
- If more than two internal standard criteria fails low < -50%), sample must be re-analyzed. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with 18 (IS low, data is likely to show high bias).
- If one or two internal standard criteria fails low < -50%), corrective action is necessary which includes qualifying compounds with associated internal standard with 18 (IS low, data is likely to show high bias).

STATE NOTE: OHIO VAP: The analyst checks the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area response for any of the internal standards changes by a factor of two (-50% to +100%) as per section 8.3.6, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered:

- Check to be sure there are no errors in the internal standards preparation or addition. Also check instrument performance.
- If any internal standard criteria fails high (> +100%), **the sample must be re-analyzed undiluted, unless obvious matrix interference is noted.** If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J9 (IS high, data is likely to show low bias).
- If any internal standard criteria fails low < -50%), **the sample must be re-analyzed sample must be re-analyzed undiluted, unless obvious matrix interference is noted.** If recoveries are still outside control limits, corrective action is necessary which

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includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).

- 11.12 CALIBRATION RANGE: The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.
- 11.13 SECOND SOURCE: The second source calibration verification standard must be analyzed following each new initial calibration to verify the validity of the calibration standards. The recovery of the analytes in the SSCV must be within 30% of the expected concentration for CCC and SPCC compounds and within 40% for non-CCC/SPCC compounds. Poor performers listed in section 8.3.4 must recover within in-house calculated LCS recovery acceptance limits. If the SSCV does not meet acceptance criteria, if can be reanalyzed once. If the failure persists, a new initial calibration curve must be prepared and analyzed.
- 11.14 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 11.14.1 If a method blank contains an amount of target analyte, but all samples are non-detected or the samples contain analyte at a level that is greater than 10 times the level present in the blank, the data is reported with the appropriate "B" flag.
- 11.14.1.1 When comparing analyte contamination in the blank to possible analyte contamination in the field sample, utilize the sample concentration without applying the multiplier value unless the same multiplier has been applied to the quantitation of the target analytes in the blank.
- 11.14.2 If the sample surrogate is above the acceptable QC range, but the samples are non-detected for all target analytes, flag the sample with a J1 and report. If the surrogate is below the acceptable QC range, re-analyze the sample if the surrogate still fails, re-extract and re-analyze or flag data.
- 11.14.3 Matrix spike failures must be flagged with "J5" (high) or "J6" (low), when QC limits are exceeded. If there is an RPD failure, the data is flagged with a "J3".

- 11.15 Quantitation and manual integration of all QC samples and client samples must follow the procedures outlined in SOP 030215, *Manual Integration*. "Before" and "After" quantitation reports must be printed in order to verify that any manual integration is performed properly and consistently.

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- 11.16 Data must be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high calibration standard. Values falling above the high standard must be diluted and re-analyzed.
- 11.16.1 Site specific DQO's may allow the reporting of values above the upper calibration standard with an "E" qualifier indicating that the value is known to be greater than the upper calibration limit. The state of South Carolina does not accept the practice of applying "E" qualifiers.
- 11.16.2 Site specific DQO's may require values below the reporting limit but above the method detection limit be reported as "UJ" or estimated value. The reporting limit is the concentration of the lowest standard used in the calibration.
- 11.16.3 All tentatively identified compounds (TICs) are reported with a "J" qualifier for estimated value and an "N" for presumptive evidence of material. Corrective actions are described in Section 11.13.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must to be characterized and disposed of in an acceptable manner. See SOP# 030301, *Waste Disposal*.

- 12.2 See SOP #030302, *Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

- 13.1 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

14.0 REFERENCES

- 14.1 "Test Methods for Evaluating Solid Waste", EPA SW-846, Method 8000B (Revision 2, September, 1996)
- 14.2 "Test Methods for Evaluating Solid Waste", EPA SW-846, Method 8260B (Revision 2, December, 1996)
- 14.2 Code of Federal Regulations, 40, Part 136, Method 624
- 14.3 Standard Methods for the Examination of Water and Wastewater, APHA, 20th edition, Method 6200B.

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- 14.4 Policy Document, NELAC Standard, Chapter 2: Proficiency Testing Program Standard and the relevant section of NELAC Standard Chapter 5 National Environmental Laboratory Accreditation Conference

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
16	1/30/09	Technical and Quality Review and update. Updated Table 1; Clarified holding times; Updated Note in Section 7; Updated section 7.4.1 & 7.4.2; Updated section 7.6, 7.8.4, 8.1.2, 8.2.3, 8.2.3.2, 8.2.4.1, 8.2.6.2 & 12.0; Added state note (MN) section 8.2.2, added final bullet item, added state note (OH); Added section 8.2.6.4; section 9.11 (state note), section 11.1 (state note), section 11.7.1 (state note), section 11.13, & 13.1; Ohio VAP approved 1/30/09

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	8/23/94	Origination
1	7/25/95	
2	9/12/97	
3	8/4/98	
4	2/11/00	
5	8/21/00	
6	4/1/01	
7	10/16/01	
8	8/19/02	
9	7/23/03	
10	10/30/03	
11	1/26/04	
12	6/28/04	
13	12/11/04	
14	3/23/05	
15	12/19/07	

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Attachment II: STATE/AGENCY SPECIFIC CRITERIA

SOUTH CAROLINA

- Specific LCS Criteria: Sections 8.3.3, 11.6
Table 11.7 footnote to LCS column
- Specific MS Criteria: Table 11.7 footnote to MS column
- LCS/MS Standards: Table 11.7 lists the target analytes that are found in the LCS and MS standard solutions
- Qualifiers: Disallows the use of "E" qualifiers
Section 8.3.3 disallows LCS failure and therefore "J4" qualifiers
- Sample Prep/Collection: Section 4.2 requires the use of 5035
Section 8.7.1 & 8.7.2 disallows the use of soil jar collection and preparation outside of the requirements of 5035
Section 8.7.2.1 & 8.7.2.2 disallows the use of waste dilution and mandates 5035
- Specific Product Codes: Non-Preserved VOC's with required 7-day Holding Time and LCS Criteria of 70-130%
V8260SC, V8260AP1SC, V8260AP2SC, V8260AP9SC, V624SC

OHIO VAP

- Spike Comp./Evaluation: Section 7.8.4 lists all required spike compounds
- Specific LCS Criteria: Sections 8.3.3, 11.6
- Specific MS Criteria: Section 10.7
- Sample Prep/Collection: Section 4.2 requires the use of 5035
Section 8.7.1 & 8.7.2 does not allow the use of soil jar collection and preparation outside of the requirements of 5035

USACE

Linear Regression Criteria of 0.995: Sections 8.2.6.3, 11.3.3

All Target Analytes required to be present and evaluated in the LCS: Section 7.4.2

Specific LCS Criteria: 11.6

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USACE (continued)

Specific MS Criteria: 11.8

Non-CCC Percent Drift Criteria: Section 8.3.2

Sample Prep Criteria: Section 8.5.5

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**Attachment III: Characteristic Masses (m/z) for Purgeable Organic Compounds
as printed from SW-846 Method 8260B Table 5**

Compound	Primary Characteristic Ion	Secondary Characteristic
		Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)

Compound	Primary Characteristic	Secondary Characteristic
	Ion	Ion(s)
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d4	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
Hexane	57	86, 56
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49

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Compound	Primary Characteristic	Secondary Characteristic
	Ion	Ion(s)
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethylene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene-d6	84	83
Bromobenzene-d5	82	162
Bromoform-d2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115, 150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174, 176

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Compound	Primary Characteristic	Secondary Characteristic
	Ion	Ion(s)
Chloroform-d1	84	
Dibromofluoromethane	113	
4-Bromofluorobenzene	95	174, 176
Chloroform-d1	84	
Dibromofluoromethane	113	
Dichloroethane-d4	102	
Toluene-d8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

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Attachment IV: Potential Compounds to be Analyzed by this Procedure

Acetone	1,4-Difluorobenzene (I.S.)
Acetonitrile	1,4-Dioxane
Acrolein	Epichlorohydrin
Acrylonitrile	Ethanol
Allyl alcohol	Ethylbenzene
Allyl chloride	Ethylene oxide
Benzene	Ethyl methacrylate
Benzyl chloride	n-Hexane
Bromoacetone	2-Hexanone
Bromochloromethane (I.S.)	2-Hydroxypropionitrile
Bromodichloromethane	Iodomethane
4-Bromofluorobenzene (Surr.)	Isobutylalcohol
Bromoform	Malononitrile
Bromomethane	Methacrylonitrile
2-Butanone	Methylene chloride
Carbon disulfide	Methyl iodide
Carbon tetrachloride	Methyl methacrylate
Chloral hydrate	4-methyl-2-pentanone
Chlorobenzene	Pentachloroethane
Chlorobenzene d-5 (I.S.)	2-picoline
Chlorodibromomethane	Propargyl alcohol
Chloroethane	B-propiolactone
2-Chloroethanol	Propionitrile
bis-(2-Chloroethyl) sulfide	n-Propylamine
2-Chloroethyl vinyl ether	Pyridine
Chlorofrom	Styrene
Chloromethane	1,1,1,2-Tetrachloroethane
Chloroprene	1,1,2,2-Tetrachloroethane
3-Chloropropionitrile	Tetrachloroethene
1,2- Dibromo-3-chloropropane	Toluene
1,2-Dibromoethane	Toluene d-8 (surr.)
Dibromomethane	1,1,1-Trichloroethane
1,4-Dichloro-2-butene	1,1,2-Trichloroethane
dichlorodifluoromethane	Trichloroethene
1,1-Dichloroethane	Trichlorofluoromethane
1,2-Dichloroethane	1,2,3-Trichloropropane
1,2-Dichloroethane d-4 (surr.)	Vinyl acetate
1,1-Dichloroethene	Vinyl chloride
Trans-1,2-dichloroethene	Xylene (total)
Cis-1,2-dichloroethene	1,2,3,4-Diepoxybutane
1,2-dichloropropane	
1,3-Dichloro-2-propanol	

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)

Attachment V: The SIM Mode:

An alternate way of running compounds to achieve lower detection limits is by way of the Single Ion Monitoring (SIM) method. The SIM method allows the Mass spec to dwell on certain ions rather than scanning the full range of masses from 35 to 300. This process allows for much lower detection limit of desired compounds. This method is only for the detection of known compounds while a TIC cannot be performed while running the SIM method. Currently 1,4-Dioxane is the only compound that is analyzed using the SIM method at ESC.

STATE NOTE: This procedure is not required for Ohio VAP samples as action levels for this analyte within the Ohio VAP standards are easily achievable using the normal SCAN mode.

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**ENVIRONMENTAL SCIENCE CORP.
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Number: 330363
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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)

Attachment VI: EPA 624 CCC Criteria:

TABLE 5—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 624^a

Parameter	Range for Q ($\mu\text{g/L}$)	Limit for s ($\mu\text{g/L}$)	Range for X ($\mu\text{g/L}$)	Range for P, P_s (%)
Benzene	12.8–27.2	6.9	15.2–26.0	37–151
Bromodichloromethane	13.1–26.9	6.4	10.1–28.0	35–155
Bromoform	14.2–25.8	5.4	11.4–31.1	45–169
Bromomethane	2.8–37.2	17.9	D–41.2	D–242
Carbon tetrachloride	14.6–25.4	5.2	17.2–23.5	70–140
Chlorobenzene	13.2–26.8	6.3	16.4–27.4	37–160
Chloroethane	7.6–32.4	11.4	8.4–40.4	14–230
2-Chloroethylvinyl ether	D–44.8	25.9	D–50.4	D–305
Chloroform	13.5–26.5	6.1	13.7–24.2	51–138
Chloromethane	D–40.8	19.8	D–45.9	D–273
Dibromochloromethane	13.5–26.5	6.1	13.8–26.6	53–149
1,2-Dichlorobenzene	12.6–27.4	7.1	11.8–34.7	18–190
1,3-Dichlorobenzene	14.6–25.4	5.5	17.0–28.8	59–156
1,4-Dichlorobenzene	12.6–27.4	7.1	11.8–34.7	18–190
1,1-Dichloroethane	14.5–25.5	5.1	14.2–28.5	59–155
1,2-Dichloroethane	13.6–26.4	6.0	14.3–27.4	49–155
1,1-Dichloroethene	10.1–29.9	9.1	3.7–42.3	D–234
trans-1,2-Dichloroethene	13.9–26.1	5.7	13.6–28.5	54–156
1,2-Dichloropropane	6.8–33.2	13.8	3.8–36.2	D–210
cis-1,3-Dichloropropene	4.8–35.2	15.8	1.0–39.0	D–227
trans-1,3-Dichloropropene	10.0–30.0	10.4	7.6–32.4	17–183
Ethyl benzene	11.8–28.2	7.5	17.4–26.7	37–162
Methylene chloride	12.1–27.9	7.4	D–41.0	D–221
1,1,2,2-Tetrachloroethane	12.1–27.9	7.4	13.5–27.2	46–157
Tetrachloroethene	14.7–25.3	5.0	17.0–26.6	64–148
Toluene	14.9–25.1	4.8	16.6–26.7	47–150
1,1,1-Trichloroethane	15.0–25.0	4.6	13.7–30.1	52–162
1,1,2-Trichloroethane	14.2–25.8	5.5	14.3–27.1	52–150
Trichloroethene	13.3–26.7	6.6	18.6–27.6	71–157
Trichlorofluoromethane	9.6–30.4	10.0	8.9–31.5	17–181
Vinyl chloride	0.8–39.2	20.0	D–43.5	D–251

^aQ= Concentration measured in QC check sample, in $\mu\text{g/L}$ (Section 7.5.3).

^s= Standard deviation of four recovery measurements, in $\mu\text{g/L}$ (Section 8.2.4).

X= Average recovery of four recovery measurements, in $\mu\text{g/L}$ (Section 8.2.4).

P, P_s = Percent recovery measured, (Section 8.3.2, Section 8.4.2).

D= Detected; result must be greater than zero.

*Criteria were calculated assuming a QC check sample concentration of 20 $\mu\text{g/L}$.

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SOP/DOC# 330363 Current revision date & number: 1/30/09 R16
Procedure/Method : VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)

*Comments:

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Add to section 7.4.1:

AP9/Oxygenates ICV Standard in 25mL total volume of methanol

Manufacturer	Product	Cat #	Amount Added	Final Concentration (PPM)
Ultra	Custom SC Oxygenates	CUS-8674	0.0625 mL	50
NSI	Custom AP9 Standard	Q-4973	3.125 mL	5

Add to section 7.4.2:

AP9/Oxygenates Second Source Standard in 25mL total volume of methanol

Manufacturer	Product	Cat #	Amount Added	Final Concentration (PPM)
Ultra	Custom AP9 Standard	CUS-9324	3.125 mL	5

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Add to section 8.2.3.1:

AP9/Oxygenates Calibration Curve – GC/MS Soil (into 5mL of water)	
Intermediate solution volume (uL)	Concentration of standard (ppb)
1	1
2.5	2.5
5	5
7.5	7.5
10	10
12.5	12.5
15	15
17.5	17.5
20	20

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Add to section 8.2.3.2:

AP9/Oxygenates Calibration Curve – GC/MS water (into 5mL of water)	
Intermediate solution volume (uL)	Concentration of standard (ppb)
1	1
2.5	2.5
5	5
7.5	7.5
10	10
12.5	12.5
15	15
17.5	17.5
20	20

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Add to section 8.2.3.3:

Calibration Curve – GC/MS water (into 50mL of water)	
Intermediate solution volume (uL)	Concentration of standard (ppb)
10	1
25	2.5
50	5
75	7.5
100	10
125	12.5
150	15
175	17.5
200	20

Add State Note to section 8.2.6.4:

STATE NOTE: For South Carolina samples, quadratic curve fitting is only allowed for analytes that do not historically exhibit a linear response. This calibration model may not be utilized to extend the calibration range or bypass instrument maintenance. Quadratic regression is not allowed for Oxygenate Compounds that have historically demonstrated linearity.

Add Additional State Note following section 9.11 to read:

STATE NOTE: For South Carolina samples, quadratic curve fitting is only allowed for analytes that do not historically exhibit a linear response. This calibration model may not be utilized to extend the calibration range or bypass instrument maintenance. Quadratic regression is not allowed for Oxygenate Compounds that have historically demonstrated linearity.

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10/25/05 R.0

SOP/DOC# VOC by GC/MS 330363 Current revision date & number: 01/30/09 R16

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10/25/05 R.0

SOP/DOC# <u>VOC by GC/MS 330363</u> Current revision date & number: <u>01/30/09 R16</u>						
Procedure/Method :						
Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
08/25/09	BW 120	11.11	Client Note: For Marathon, the internal standard area counts for all calibration standards, QC samples, and samples for quantitation must not change by a factor of greater than (-50% to +130%) as per section 8.3.6.	CAR 781	BW 120	DLM

[Handwritten notes and signatures:]

A handwritten note is present in the blank rows of the table, reading: "Client Note: For Marathon, the internal standard area counts for all calibration standards, QC samples, and samples for quantitation must not change by a factor of greater than (-50% to +130%) as per section 8.3.6." There is also some faint handwriting below the note, including what appears to be initials and a date.

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10/25/05 R.0

SOP/DOC# <u>8260_624</u> <u>330363</u>	Current revision date & number: <u>01/30/09 R17</u>
Procedure/Method : 8260	

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
10/01/09	BW 120	8.2.2	8260C note - The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.	Additions for 8260C	<i>BW</i> <i>120</i>	<i>JAM</i>
		8.2.6. 1	8260C note – The %RSD criteria for each individual CCC does not apply for 8260C.			
		8.2.6. 2	8260C - <20%RSD for all 8260C Target Analytes			

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		8.2.6	8260C note - Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet either the 20% RSD, minimum correlation coefficient criteria (0.99), or the acceptance criteria for alternative calibration procedures in Method 8000. Any calibration method stipulated in Method 8000 may be used, but it should be used consistently. It is considered inappropriate once the calibration analyses are completed to select an alternative calibration procedure in order to pass the recommended criteria on a case by case basis. If compounds fail to meet these criteria, the associated concentrations may still be determined but they must be reported as estimated. In order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable lower quantitation limit.				
		8.2.6. 5	8260C note - When the RSD exceeds 20% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.				
		8.3.2	8260C note - All target compounds of interest must be evaluated using a 20% variability criterion. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.				
		10.3/ 10.4/ 10.5	Include 8260C.				
		8.3.2	8260C note - If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, some compounds will fail to meet the criteria. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.				

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		8.3.3	8260C note - Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum response factor for the most common target analytes as noted in 8.2.5, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet this criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes.			BW DM 120	
		8.2.5	Insert attached table 4 for 8260C Min RF's.				
		8.3.5	8260C note - A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Also note the LCS for water sample matrices is typically prepared in organic-free reagent water similar to the continuing calibration verification standard. In these situations, a single analysis can be used for both the LCS and continuing calibration verification.				

*Comments: _____

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND
CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

^a The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

^b Data provided by EPA Region III laboratory.

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Procedure/Method : VOC by GC/MS (8260/624)	

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
10/30/09	BW 120	8.3.4	Add Pentachloroethane and Cyclohexanone to the list of poor performers.		<i>BW 120</i>	<i>D</i>

*Comments: _____

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SOP/DOC# 330363 Current revision date & number: R16 1/30/09

Procedure/Method : **VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 624 AND SM6200B 20TH)**

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
10/7/11	DLM	11.7.1	Revise the first line in the number of allowable marginal excedences to reflect 90+ analytes, 5 analytes allowed in the ME limit.	correction	JDG	DML

*Comments:



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SOP Revision Summary

SOP:			
Author - Shakir Wani/Chris John	Number - 330345	Department - SVOC	
Title - SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM	F		
Revision - R13	Rev. Date - 11/23/2010		

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
1	12/10/2010	Various	Inclusion of all relevant sections for extraction of samples using LVI (EPA 3511) for PNAs and PNA by SIM.

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(see Revision History at the end of this document for more information)

TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

SOP NUMBER: 330345

Prepared by: Shakir Wani/Chris Johnson

Reviewed by: Chris Johnson/Blake Judge/Dixie Marlin

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Department Manager

QA Department

1.0 SCOPE AND APPLICATION

- 1.1 This method is used to determine the concentration of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. The lists of compounds that are routinely determined by this method are listed in Attachment II. This table represents a default list to be used in the absence of a project-specific list, which would take precedence. See section 13.4.
- 1.2 This method is used to quantitate most neutral, acidic and/or basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica column coated with a slightly polar methyl silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.
- 1.3 In general, this method is not appropriate for the quantitation of multi-component analytes (i.e. Toxaphene, Chlordane, Aroclors, etc.) because of the limited sensitivity for those analytes; however when those analytes are identified using another analytical technique, this procedure is appropriate for confirmation pending sufficient analyte concentration is present in the extract.
- 1.4 Detection limits, sensitivity and optimum ranges of organic compounds vary with sample matrices, extraction technique, detector parameters, and model of GC/MS.
- 1.5 Qualifier ions are method specified and can be found in Attachment V.

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

- 1.6 Use of this method is restricted to analysts who are knowledgeable in the interpretation of Mass Spectrometry and use of GC/MS systems.
- 1.7 The use of selected ion monitoring (SIM) is acceptable for applications requiring limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.
- 1.8 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 Field samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample extraction technique. See ESC SOPs 330702/330702A/330705/330707/330708/330709/330754 for extraction and extract concentration methods.
- 2.2 The semi-volatile compounds are introduced into the GC/MS by directly injecting a volume of the sample extract into a gas chromatograph oven (GC) equipped with a narrow-bore fused-silica capillary column. The oven, containing the capillary column, is temperature and pressure programmed to separate the analytes by molecular composition. The capillary column transfers the eluting analytes to the detector (MS) connected to a computer that then collects and stores the information for each injection.
- 2.3 Identification of target analytes is accomplished by comparing the mass spectra of each peak with the reference spectra of authentic standards.
- 2.4 Quantitation of the analytes of interest is accomplished by comparing the response of a major (quantitation) ion, present in the target analyte, relative to an internal standard in each extract, in conjunction with the response factor generated from a calibration curve.
- 2.5 Proper quantitation ions for each compound must be selected so that no interferences are present from adjoining (or co-eluting) analytes with common ions. Proper GC conditions must be used to resolve compounds with similar mass spectra. Background subtraction of mass spectra may be necessary when matrix interference is present.

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

- 2.6 Qualitative - The identification of compounds based on retention time and comparison of the sample mass spectra, after background correction, with characteristic ions in the reference mass spectra. The reference mass spectra must be generated by the laboratory using the same analytical conditions used for the analysis of field samples. The characteristic ions from the reference mass spectra are defined as the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectra.
- 2.7 Quantitative – Following qualitative identification, the quantitation of the identified compound is based on the integrated abundance of the primary characteristic ion from the Extracted Ion Current Profile (EICP).
- 2.8 Tentatively Identified Compound (TIC) – Non-calibrated analytes that are, minimally, 10% of the response of the nearest internal standard. These peaks are tentatively identified using the comparison of the spectra of the peak in question to spectra in the mass spectral library for possible matches.
- 2.9 Initial Demonstration of Capability (IDOC) - A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank, laboratory control sample, etc. or the demonstration of capability must be repeated. See also Continuing Demonstration of Capability (CDOC).
- 2.10 Continuing Demonstration of Capability (CDOC) – At least annual verification of analyst continued ability to perform method acceptably.
- 2.11 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) – Duplicate aliquots of a control sample of known composition. This sample is prepared from a source that is different from the stock used to prepare the initial and continuing calibration standards. LCS/LCSD are analyzed exactly like a sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Method precision can be determined using the results of the LCS/LCSD analysis.
- 2.12 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.

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- 2.13 Method Blank - An analytical control consisting of all reagents used in the analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.
- 2.14 Method Detection Limit (MDL) - The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero.
- 2.15 Reporting Limit (RL) – Also see Practical Quantitation Limit (PQL). Routinely the reporting limit is the lowest standard of the calibration curve. Technically, the reporting limit is the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions.
- 2.16 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the MDL.
- 2.17 Second Source Calibration Verification (SSCV) – A mid-point or low standard made from a secondary standard that is not used to construct the calibration curve. The SSCV is used to represent the calibration accuracy of the instrument and must perform within method stated criteria.
- 2.18 Internal Standard (ISTD) – Analytes not expected to occur naturally in field samples that are spiked to provide a consistent basis for use in internal calibration models.
- 2.19 Internal Calibration – Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific spikes added to the sample or sample extract prior to injection.
- 2.20 Response Factor (RF) - The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the relevant internal standard in the sample or sample extract.
- 2.21 Relative Response Factor (RRF) – The Response Factor (RF) calculated relative to the response factor of the internal standard.
- 2.22 Sample Extraction - A sample of a known volume or weight is prepared for analysis by removing soluble substances using solvent.
- 2.23 Surrogate - A compound, similar to the target analytes in chemical composition and behavior, but not expected to occur naturally in field samples. Analytes are spiked by preparation/analytical personnel to assess sample extraction and analytical efficiency in each individual field sample.

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- 2.24 Elution - The order of emergence of chemicals from the column of a chromatograph. The chemicals then typically flow into a detector of some type. Predicting and controlling the order of elution is a key aspect of column chromatographic methods and can be modified using instrument operating conditions, column selections, etc.
- 2.24.1 Co-elution – Peaks that are not distinctly separated or resolved by a chromatograph. Co-elution is problematic when peaks share primary and secondary mass ions making accurate quantitation questionable.
- 2.25 Retention Time – The expected time that it takes for a particular analyte to pass through the system (from the column inlet to the detector) under set conditions.
- 2.26 External Calibration - External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF). See DRO modification for Kansas sample analysis.
- 2.27 Reporting Limit Verification (RLV) – A standard analyzed following initial calibration/calibration verification at or below the analyte concentration of the routine reporting level. It is analyzed per regulatory/method requirements for drinking water analyses and various other state/national regulatory programs to verify the accuracy of field sample results at the reporting level.
- 2.28 Calibration Standards – Solutions of known concentrations used to create graphic representation of the relationship between the known values, such as concentrations and instrument responses.
- 2.29 Linear Regression - Mathematical technique for finding the straight line that best-fits the values of a linear function, plotted on a scatter graph as data points. If a 'best fit' line is found, it can be used as the basis for estimating the future values of the function by extending it while maintaining its slope.
- 2.30 Quadratic Regression - Mathematical technique for finding the parabolic line that best-fits the values plotted on a scatter graph as data points. If a 'best fit' line is found, it can be used as the basis for estimating the future values of the function.

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3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical must be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets must also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.
- 3.2 **CAUTION:** Be careful when diluting and mixing acids. ALWAYS pour acid into water when mixing. Gently heat acid mixtures (NEVER HEAT RAPIDLY), to prevent splatter from extremely exothermic reactions typical of acid-water mixtures, etc.
- 3.3 Prior to performing this procedure, the analyst should be familiar with the proper use of corrosive liquid spill kits and contaminant procedures.
- 3.4 Much of the instrumentation used in this procedure has heated zones that can cause severe burns. Always unplug all instruments before doing any maintenance that involves electrical parts.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Requirements for sample extraction are detailed in SOP numbers 330702, 330702A, 330705, 330707, 330708, 330709, and 330754.
- 4.3 The sample holding time for solid samples is 14 days to extraction and, for aqueous samples, the holding time is 7 days. Holding time begins when (date and time) the samples are collected and ends either 14 or 7 days following sampling, at the time sampled.
- 4.4 The holding time for each extract is 40 days from sample preparation to analysis.
- 4.5 The container for aqueous samples and liquid sludge are 1L amber glass bottles. Add 0.008% Na₂S₂O₃ per liter, if residual chlorine is expected or present.

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- 4.6 Collect solid sample materials in 4 oz. jars or larger, depending on the weight and density of the sampled materials.
- 4.7 All samples and extracts must be shipped and stored at <6°C.

5.0 INTERFERENCES

- 5.1 Raw GC/MS data from all method blanks, samples, and spikes is evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of samples and take corrective action to eliminate the problem.
- 5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of solvent to check for cross-contamination. Clean/replace injector liner or clip column, check with solvent blanks, and repeat samples if necessary.
- 5.3 Choice of quantitative ions and qualifier ions: Some compounds may co-elute, so the selection of quantitation ions and qualifier ions must be made carefully so these ions are specific to each of the compounds that co-elute. Qualifier ions that are most commonly used are listed in Attachment V and are recommended from the published 8270 methods. ESC uses these ions as recommended by the method with exception of the following: 2,4-Dimethylphenol, 2-Methylphenol, 2-Nitroaniline, 4-Nitrophenol, Acenaphthene, Benzoic Acid, Benzyl Alcohol, bis(2-Chloroisopropyl)ether. There is no method stated ions for the following: Pyridine, 1-Methylnaphthalene, Biphenyl, Carbazole. Refer to Attachment V for ESC ions.
- 5.4 Problematic Compounds:
 - 5.4.1 Benzidine may be subject to oxidative losses during solvent concentration and exhibits poor chromatographic behavior.
 - 5.4.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the GC inlet, as well as photochemical decomposition.
 - 5.4.3 N-nitrosodimethylamine may be difficult to separate from the solvent using the chromatographic conditions listed in this method.
 - 5.4.4 N-nitrosodiphenylamine decomposes in the GC inlet and can't be separated from diphenylamine.

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- 5.4.5 Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, 4,6-Dinitro-2-methylphenol, 4-Chloro-3-methylphenol, 2-Nitroaniline, 3-Nitroaniline, 4-Chloroaniline, and Benzyl Alcohol are subject to erratic chromatographic behavior, especially when there is high boiling material contamination of the GC system.
- 5.4.6 Pyridine may perform poorly at the GC injection port temperatures listed in this method. The amount of degradation may be reduced by lowering the injection port temperature. Modification of the injection port temperature may adversely affect the performance of other target analytes.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Gas chromatograph/mass spectrometer system.
 - 6.1.1 Gas chromatograph (HP 6890 or equivalent)- An analytical system complete with a temperature- programmable gas chromatograph suitable for split-less injection and all required accessories, including, auto sampler, syringes, analytical columns, and gases. The capillary column is directly coupled with the source.
 - 6.1.2 Column 1 - 30m x 0.25mm ID with a 0.25µm film thickness silicon-coated fused silica capillary column (Phenomenex ZB-5MS or equivalent).
 - 6.1.3 Column 2 – J&W 30m x 0.25mm x 0.5um film DB5MS or an equivalent is used. Ultra pure (99.999%) Helium gas is used for a mobile phase.
 - 6.1.4 Syringes: Agilent (or equivalent) syringes sizes 10µL, 25µL, 50µL, 100µL and 1.0mL.
- 6.2 Mass spectrometer (HP-5973/5975 or equivalent). Capable of scanning from 35 to 550 amu every 1 second, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrum for decafluorotriphenylphosphine (DFTPP) must meet the criteria in method 8270C when 1-2µL of the GC/MS tuning standard is injected (50ng of DFTPP)
- 6.3 GC/MS interface - The interface is capillary-direct into the mass spectrometer source.

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- 6.4 Data system (HP Chemstation with Enviroquant) - A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage of machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as Extracted Ion Current Profile (EICP). The most recent version of the EPA/NIST Mass Spectral Library is also available
- 6.5 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.
- 6.6 Balance - Analytical, capable of weighing 0.0001g

7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner if a problem is detected unless otherwise noted.
- 7.2 Reagent grade inorganic chemical are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 7.3 Organic-free reagent water - all references to water in this method refer to organic-free reagent water (ASTM II or equivalent).
- 7.4 Burdick & Jackson Omni Solv Dichloromethane Dx0831-1 (or equivalent)
- 7.5 Stock standard solutions - Standard solutions are purchased as certified solutions. Commercially-prepared stock standards are used at concentrations that are certified by the manufacturer or by an independent source. A stock standard is purchased from Ultra Scientific with all targets at 200µg/mL; use this directly from the vial with no dilution.
 - 7.5.1 Restek, Custom 8270 Mix – 56321, or equivalent, at 200ppm
 - 7.5.2 NSI, 8270 TCL Project Mix – Q4296, or equivalent, at 1000ppm
 - 7.5.3 AccuStandard, Composite Mix #3 – Z-014E-R3, or equivalent, at 2000ppm

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- 7.5.4 Restek, Benzoic Acid Mix – 31879, or equivalent, at 2000ppm
- 7.5.5 Restek, Benzidine Mix #2 – 31852, or equivalent, at 1000ppm
- 7.5.6 AccuStandard, 2-Nitrodiphenylamine – S-4829A, or equivalent, at 200ppm
- 7.5.7 B/N Surrogate Mix – C-376M-39, or equivalent, at 2000ppm
- 7.5.8 Organic Acid Surrogate Mix – C-131M-24, or equivalent, 4000ppm
- 7.5.9 Second Source: Restek, 8270 MegaMix – 31850, or equivalent, at 1000ppm
- 7.5.10 Transfer the stock standard solutions into bottles with PTFE-lined screw caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standards are assigned a 6 month expiration date from the day that a sealed ampoule is opened. Standards are discarded if signs of degradation are apparent when compared to a second source standard.
- 7.6 For PAHs by SIM, use a custom mix purchased from Ultra Scientific (Cat#: CUS-9356) with all required PAH targets. Each target compound is at concentration of 200ppm. Other concentrations may be acceptable with dilutions as appropriate for yielding the appropriate concentrations in the secondary source. The secondary source is also a custom mix from Ultra Scientific (Cat#: CUS-9345) at 200ppm.
- 7.7 For the DROMO by GC/MS, use a custom mix purchased from Ultra Scientific (Cat#: CUS-8255), or equivalent, which is a neat solution of diesel and an Ultra Scientific custom mix (Cat#: CUS-8254) for the gasoline components at neat. Alternatively, calibration standards can be prepared using the TX TPH Calibration Mix from Restek (Cat#: 31483) at 10,000ppm each. The secondary source is an NSI, Diesel Range Organic Spike (Cat#: Q4394) at 2500ppm each.

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- 7.8 Internal standards solutions- the internal standards are naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, perylene-d12 and 1-4 dichlorobenzene-d4. Purchase from AccuStandard (Cat #: A-014J-PAK) as certified stock solution at 4000ppm. Purchase from NSI (Cat # Q-6343-O) as certified stock solution at 800ppm.
- 7.8.1 For SIM, PAH & BNA, use the 800 μ g/mL solution. Each 1.0mL of sample extract undergoing analysis is spiked with 10 μ L of internal standard intermediate solution, resulting in a concentration of 8 μ g/mL of each internal standard.
- 7.8.2 For DROMO and AP9/AP2 analyses use the 4000 μ g/mL solution. Each 1.0mL of sample extract undergoing analysis is spiked with 10 μ L of internal standard intermediate solution (or adjusted for AP9/AP2) to result in a concentration of 40 μ g/mL of each internal standard.

7.9 Preparation of Intermediate Standard

Stock Mix	Section ID:	Amount Added (mL)	Concentration of Stock in ppm	Concentration in Intermediate (ppm)
Restek - Custom Mix with Surrogates	7.5.1	--	200	--
NSI - 8270 TCL Project Mix	7.5.2	2.0	1000	200
AccuStandard - Composite Mix #3	7.5.3	1.0	2000	200
Restek - Benzoic Acid Mix	7.5.4	1.0	2000	200
Restek - Benzidine Mix #2	7.5.5	1.0	2000	200
AccuStandard - 2-Nitrodiphenylamine	7.5.6	2.0	1000	200

Using a volumetric syringe, measure each of the solutions listed in Section 7.9 and place into a 10mL volumetric flask. The final concentration will be 200 μ g/mL of each component. Use this solution or the certified custom mix purchased from Restek in section 7.5.1 to prepare the working standards in Table 7.10.

- 7.9.1 For SIM, prepare a 5 μ g/mL intermediate by diluting the 10 μ g/mL PAH mix described in section 7.6.

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7.10 Preparation of Working Standards

Standards must be stored at 4 ± 2°C. The expiration date of any working standard will be 6 months unless the stock expires prior to that date or if the standard starts showing degradation. See Table 7.10.1 for preparation instructions. Concentration of standards used is subject to change depending on instrument condition and/or client needs. A minimum of five calibration levels is required for Method 8270, while a minimum of 3 calibration levels is required for Method 625.

7.10.1 Calibration standards: A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system. Each standard contains each analyte for detection. Working standards are made directly from the Ultra scientific certified stock standard described in section 7.5 to give solutions at concentrations of 0.2µg/mL up to 100µg/mL. Surrogates are included at the same concentration. Internal standards are at a constant at 8 or 40µg/mL for quantitation purposes.

TABLE 7.10.1
SVOC Initial Calibration (5-point)

SVOC mix (200ppm) µL	ISTD mix µL	Final volume	Final conc. ppm	Level
1	10	1.0mL	0.2	1
5	10	1.0mL	1	2
10	10	1.0mL	2	3
20	10	1.0mL	4	4
40	10	1.0mL	8	5
60	10	1.0mL	12	6
80	10	1.0mL	16	7
100	10	1.0mL	20	8
200	10	1.0mL	40	9
400	10	1.0mL	80	10

A minimum of 5 points are used to construct the calibration curve.

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- 7.10.2 For SIM, calibration standards are diluted from the intermediate standard solution (section 7.9.1) to give a calibration at the following concentrations: 20, 50, 100, 500, 1000, 2000, 4000, 10,000 μ g/L. A minimum of five calibration levels from the above list is analyzed. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The calibration levels may change based on the working range of the GC/MS system. The internal standards are at a constant 8 μ g/mL.

SIM Standard Concentration (ug/L)	Amount Added (uL)	Final Volume (mL)
20	2.0	1.0
50	5.0	1.0
100	10.0	1.0
500	50.0	1.0
1000	100.0	1.0
2000	200.0	1.0
4000	400.0	1.0
10000	1000.0	1.0

- 7.10.3 For SIM, prepare a 1.0 μ g/mL surrogate standard. Add a 1.0mL to samples QC (method blanks, MS/MSD and LCS/LCSD) prior to extraction. Concentration will be 1 μ g/mL in the extract. Matrix spike and laboratory control sample standards are from a different source or a different lot number than the calibration. Prepare a PAH matrix spiking solution at 2.0 μ g/mL. Add 1.0mL to spikes (MS/MSD/LCS/LCSD) prior to extraction. Concentration will be 2ppm in the extract.
- 7.11 DFTPP Standard Prep for 50ppm Solution – 50 μ L of 1000 ppm DFTPP (AccuStandard M-625-TS-20X) + 950 μ L of Methylene Chloride (final volume of 1mL).
- 7.12 DFTPP Standard Prep for 25ppm Solution – 25 μ L of 1000 ppm DFTPP (AccuStandard M-625-TS-20X) + 950 μ L of Methylene Chloride (final volume of 1mL).
- 7.13 Surrogates and Spike Solutions - Preparation techniques can be found in the appropriate extraction technique for the given matrix. See SOP No. 330702, *Separatory Funnel Liquid-Liquid Extraction (3510B)* or SOP No. 330707, *Microwave Extraction (3546)* for soil/solid samples.
- 7.14 See section 13.4 for additional information regarding standards and spiking solutions.

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8.0 PROCEDURE

STATE NOTE: For samples analyzed in conjunction with the Ohio VAP program, the criteria found and itemized in this procedure for EPA method 8270C must be utilized.

8.1 GC Conditions: The GC conditions are listed in each instrument maintenance log and are updated as necessary.

8.2 Mass Spectrometer Tuning Criteria:

The GC/MS is hardware-tuned using a 50ng injection of DFTPP. Analyses must not begin until the tuning criteria in Table 8.2a/b are met. The following options are available for acquiring the spectra for reference to meet the DFTPP tuning requirements. It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at three scans (the apex & ± 1 scan) and average the three scans.

Background subtraction is required prior to the start of the peak but no more than 20 scans prior. Background correction cannot include any parts of the target peak. The entire peak may be averaged and background corrected. Average scans 0.1 minute before to 0.1 minute after the target peak including the peak apex. The mass spectrometer must be tuned every 12 hours if samples, standards, etc. are to be analyzed for Method 8270C or 8270D or every 24 hours for Method 625.

TABLE 8.2a
Method 8270C/625/SM6410B:
DFTPP Key Ions And Ion Abundance Criteria^(a, b)

Mass Ion Abundance Criteria	
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but < mass 442
442	>40% of mass 198
443	17-23% of mass 442

(a) Data taken from Reference 3 in SW-846 Method 8270C.

(b) Alternate tuning criteria may be used (e.g., CLP, Method 525, or manufacturers' instructions), providing that method performance is not adversely affected.

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TABLE 8.2b
Method 8270D:
DFTPP Key Ions And Ion Abundance Criteria^(a, b)

Mass Ion Abundance Criteria	
51	10-80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	10-80% of mass 198
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5-9% of mass 198
275	10-60% of mass 198
365	>1% of mass 198
441	Present, but <24% of mass 442
442	Base peak, or >50% of mass 198
443	15-24% of mass 442

(a) Data taken from Table 3 in SW-846 Method 8270D.

(b) Alternate tuning criteria may be used (e.g., CLP, Method 525, or manufacturers' instructions), providing that method performance is not adversely affected.

STATE NOTE: All South Carolina samples require a tune every 12 hours, regardless of which method is being utilized.

The GC/MS tuning standard solution must also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD is used to assess breakdown occurring in the injection port. The calculation for the determination of the breakdown occurring is found in section 9.1 and must include both DDD and DDE. Breakdown must not exceed 20%. Benzidine and pentachlorophenol are used to assess tailing occurring within the analytical system and both analytes should be present at their normal responses with no obvious peak tailing. To determine the tailing factor for benzidine and pentachlorophenol, use the calculation found in section 9.2. For EPA Methods 625 and 8270C, benzidine must have a tailing ratio of <3 and pentachlorophenol must have a tailing ratio of <5. For EPA Method 8270D, benzidine and pentachlorophenol must have a tailing ratio of <2.

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8.3 Calibration

8.3.1 Initial Calibration

EPA Method 8270C: The working standards prepared in Table 7.10.1 are injected and average response factors are calculated. The calibration curve is typically constructed of nine standards, however, this may change depending on instrument conditions and/or client needs (see Section 13.4). In the event that a calibration point is eliminated, the analyst will make a note of explanation on the raw data.

The calibration control compounds (CCCs) listed in Section 8.3.1a must have an average RSD of less than or equal to 30%. Any target analyte that has a %RSD >15% for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is ≤15%, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or quadratic curve fit. The calibration curve cannot be forced through zero and does not include a method blank. It must also meet a correlation coefficient of 0.990 or better. Analyses being generated for USACE projects must meet a correlation coefficient of 0.995 or better. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve.

The system performance check compounds (SPCCs) in Table 8.3.1b must have an average RF of ≥0.05. When these criteria are met, samples can be analyzed.

Table 8.3.1a: Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
n-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Table 8.3.1b: System Performance Check Compounds (SPCC)

Compound	Minimum Average Response Factor
n-Nitroso-di-n-propylamine	>0.05
Hexachlorocyclopentadiene	>0.05
2,4-Dinitrophenol	>0.05
4-Nitrophenol	>0.05

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EPA Method 8270D: The working standards prepared in Table 7.10.1 are injected and average response factors are calculated. The calibration curve is typically constructed of nine standards, however, this may change depending on instrument conditions and/or client needs (see section 13.4). At least five standards are required for Response Factor and linear regression calibration. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve. In the event that a calibration point is eliminated, the analyst will provide an explanation on the raw data.

Target analytes must have an average RSD of $\leq 20\%$. Any target analyte that has a %RSD $> 20\%$ for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is $\leq 20\%$, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or, if permitted, quadratic curve fit. The calibration curve cannot be forced through zero. It must also meet a correlation coefficient of 0.990 or better. Analyses being generated for USACE projects must meet a correlation coefficient of 0.995 or better.

In addition to the minimum %RSD criteria, it is recommended that a minimum response factor for the most common target analytes be demonstrated for each individual calibration level to ensure that these compounds are performing as expected. See Table 8.3.1c. Meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

Table 8.3.1c: Recommended Minimum Response Factors for Each Calibration Level (Initial and Continuing Calibration)

Compound	Minimum Response Factor	Compound	Minimum Response Factor
Benaldehyde	0.010	2,4-Dimethylphenol	0.200
Phenol	0.800	Bis(2-chloroethoxy)methane	0.300
Bis(2-chloroethyl)ether	0.700	2,4-Dichlorophenol	0.200
2-Chlorophenol	0.800	Naphthalene	0.700
2-Methylphenol	0.700	4-Chloroaniline	0.010
2,2-Oxybis-(1-chloropropane)	0.010	Hexachlorobutadiene	0.010
Acetophenone	0.010	Caprolactam	0.010
4-Methylphenol	0.600	4-Chloro-3-methylphenol	0.200
n-Nitroso-di-n-propylamine	0.500	2-Methylnaphthalene	0.400
Hexachloroethane	0.300	Hexachlorocyclopentadiene	0.050
Nitrobenzene	0.200	2,4,6-Trichlorophenol	0.200
Isophorone	0.400	2,4,5-Trichlorophenol	0.200
2-Nitrophenol	0.100	1,1-Biphenyl	0.010

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Compound	Minimum Response Factor	Compound	Minimum Response Factor
2-Chloronaphthalene	0.800	Pentachlorophenol	0.050
2-Nitroaniline	0.010	Phenanthrene	0.700
Dimethyl phthalate	0.010	Anthracene	0.700
2,6-Dinitrotoluene	0.200	Carbazole	0.010
Acenaphthylene	0.900	Di-n-butyl phthalate	0.010
3-Nitroaniline	0.010	Fluoranthene	0.600
Acenaphthene	0.900	Pyrene	0.600
2,4-Dinitrophenol	0.010	Butyl Benzyl phthalate	0.010
4-Nitrophenol	0.010	3,3-Dichlorobenzidine	0.010
Dibenzofuran	0.800	Benzo(a)anthracene	0.800
2,4-Dinitrotoluene	0.200	Chrysene	0.700
Diethyl phthalate	0.010	Bis (2-ethylhexyl)phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010	Di-n-octyl phthalate	0.010
4-Chlorophenyl-phenyl ether	0.400	Benzo(b)fluoranthene	0.700
Fluorene	0.900	Benzo(k)fluoranthene	0.700
4-Nitroaniline	0.010	Benzo(a)pyrene	0.700
4,6-Dinitro-2-methylphenol	0.010	Indeno(1,23-c,d)pyrene	0.500
4-Bromophenyl-phenyl ether	0.100	Dibenz(a,h)anthracene	0.400
n-Nitrosodiphenylamine	0.010	Benzo(g,h,i)perylene	0.500
Hexachlorobenzene	0.100	2,3,4,6-Tetrachlorophenol	0.010
Atrazine	0.010		

GC/MS SIM: All target compounds must be treated as CCCs. All analytes must have an average RSD of $\leq 30\%$.

EPA Method 625: The working standards prepared in Table 7.10.1 are injected and average response factors are calculated. The calibration curve is typically constructed of three standards, however, this may change depending on instrument conditions and/or client needs (see Table 7.10.1). A minimum of 3 points calibration is required for method 625. The %RSD is calculated for the standards analyzed and must be $\leq 35\%$ for all compounds in order to assume linearity.

All Published Methods: Reference spectra must be updated upon analysis of each new calibration curve.

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STATE NOTE: For all Minnesota sample analyses, the RL level standard will be re-injected and quantitated against the newly updated calibration curve or will be reprocessed (re-quantitated) using the completed calibration curve and will be evaluated for the $\pm 40\%$ deviation criterion with the exception of the listed poor performers in this procedure.

STATE NOTE: For all Wisconsin sample analyses, analysts must evaluate the %RSD of calibrations to ensure that they do not have unacceptable curvature. The %RSD limit criteria, as found in the specific methods listed above, applies to calibrations using average RF calibrations. For linear and quadratic curve fits, a limit of 40%RSD is used for normal target analytes and 50%RSD is utilized for known poor performing compounds.

STATE NOTE: When analyzing samples in conjunction with the Ohio VAP and South Carolina programs, the calibration model must be RSD or linear. Quadratic curve modeling is not permitted unless historical performance of analytes exhibited a nonlinear response. Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance.

8.3.2 CALIBRATION POINTS – Usage and Deletion

When the appropriate number of calibration standards are used (see section 13.4), all points must be considered in the average response factor calculation or linear regression calculation. The deletion of the highest point is acceptable when necessary, with the analyst noting that the high end of the calibration has been lowered. The deletion of the lowest calibration point is acceptable, when necessary, provided that the analyst notes the deletion on the injection log and raises the reporting limit, if necessary, for that compound. A level in the middle of the calibration curve may be deleted in the case of instrument malfunction (for that level), such as a bad injection, provided the analyst notes this on the injection log and there are still the appropriate, method defined number of calibration points remaining for each compound for each method. In no case can a middle calibration point be partially deleted. If a middle calibration point is unsuitable for several compounds then it must be deemed unsuitable for all compounds of that level.

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8.3.3 **EPA Method 8270D:** LINEAR REGRESSION USE – The method of linear regression calibration has the potential for a significant bias to the lower portion of the calibration model. This bias is not normally seen in relative percent difference methods. When utilizing linear regression fits, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the lowest concentration standard back into the completed calibration curve. It is not necessary to re-analyze a low concentration standard, but using the analytical system software, the low standard can be re-quantitated as if it were a field sample. The recalculated concentrations of the analytes utilizing the linear regression curve fit must be within $\pm 30\%$ of the true standard concentration.

STATE NOTE: For the analysis of South Carolina samples, Hexachlorophene is required to utilize linear regression. Quadratic curve fit is not allowed. To achieve this, the calibration curve may be modified by the removal of the lowest two levels and will utilize calibration levels of 60, 80, 100, 120, and 140 for quantitation of this analyte in South Carolina samples. The reporting limit (RL) for South Carolina will routinely be 100ppb for water samples.

8.3.4 Second Source Calibration Verification – the initial calibration for each target analyte must be checked with a standard from a source that is different from those used for initial calibration.

8.3.5 **Daily Tuning and Continuing Calibration**

As with the initial calibration, the system must be tuned with 50ng of DFTPP to meet the acceptance criteria found in section 8.1. Following successful tuning, the midpoint level standard (CCV) is analyzed. Calibration verification for each method, as listed below, must be met prior to the analysis of field samples.

EPA Method 8270C: The percent difference of the CCCs (see Table 8.3.1a & b) in the mid-level standard must be $<20\%$ and the SPCCs must have an RF ≥ 0.05 . The retention time of the internal standards must be within ± 30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within -50% to $+100\%$. Once these criteria are met, samples can be analyzed.

EPA Method 625: The response for any parameter in the method from the mid-level standard must not vary by more than $\pm 20\%$ from the initial calibration curve.

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EPA Methods 8270C and 625 (analyzed concurrently): The CCV must be evaluated for CCC and SPCCs as per EPA Method 8270C requirements. All non-CCC and other target analytes must meet the criteria established in Method 625 for all analytes ($\pm 20\%$). For analytes not contained in the Method 625 analyte list, the analyst evaluates the CCV and the experience of the analyst weighs heavily in determining the usability of the data.

STATE NOTE: For all Wisconsin sample analyses, non-CCC compounds for 8270C requires a $\pm 50\%$ criteria for the CCV.

GC/MS SIM: When using the SIM method, all compounds in the CCV must be treated as CCCs and must meet the minimum requirements of $\leq 20\%$ difference.

EPA Method 8270D: Each of the most common target analytes in the CCV must meet the minimum response factors in Table 8.3.1c. When using the average RF, the percent difference for each target compound in the CCV must be $\leq 20\%$. When using regression fit calibration, the percent drift of the CCV must be $\leq 20\%$. The retention time of the internal standards must be within ± 30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within -50% to $+100\%$.

- 8.3.6 For corrective action regarding tuning and calibration, see sections 11.1 and 11.2.
- 8.4 Method Blank Analysis – A method blank should be analyzed prior to any field sample analysis to verify that the analytical system is free from contaminants. If the method blank indicates that contamination may be present in the analytical system, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts.
- 8.5 GC/MS analysis of field samples and preparation QC.
 - 8.5.1 It is highly recommended that the extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.
 - 8.5.2 Allow the extracts to warm to room temperature. Just prior to analysis, add 10 μ L of the internal standard solution to the 1mL concentrated extract or 5 μ L of the internal standard solution to the 0.5mL extract obtained from sample preparation.

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- 8.5.3 The extracts are injected into the GC/MS system via autosampler, using the same operating conditions that were used for the calibration. The volume to be injected should contain 10 μ g/mL of base/neutral and 20 μ g/mL of acid surrogates (assuming 100% recovery). If SIM analysis is performed, the volume injected should result in 1 μ g/mL of the base/neutral analytes (SIM is not performed for acid compounds) and for PAHMO/DROMO, the resulting concentration injected should be 50 μ g/mL.
- 8.5.4 If the response for any quantitation ion exceeds highest level of the initial calibration range, the extract must be diluted and re-analyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (8 or 40ng/uL, unless a more sensitive GC/MS system is being used). For example, if performing a 1:10 dilution on a concentrated extract, take 100uL of the extract and dilute to a volume of 1mL with the appropriate solvent. Add 9uL of internal standard solution to the diluted extract and inject on the analytical system. It can be assumed that 1uL of internal standard was contained in the 100uL extract used for the initial dilution.
- 8.5.5 Internal standard area counts and retention times must be monitored in all samples, spikes and method blanks to monitor system performance, check for drifting, ensure effective autosampler performance, etc. If the area of the Extracted Ion Current Profile (EICP) changes by a factor of 2 (-50% to +100%) from the areas in the daily CCV, corrective action is required. The RRT of the internal standard in the extract must be within \pm 0.06RRT units of the RRT of the daily CCV.

STATE NOTE: With each new calibration curve, a reporting limit verification (RLV) standard must be analyzed for samples analyzed from Minnesota. This standard consists of either re-injecting the low calibration standard(s) or re-processing the low standard(s) utilized in the construction of the calibration curve. The RLV must recover within \pm 40% of the expected concentration. See section 11.10 for additional information.

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8.6 Qualitative Identification

- 8.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Retention time windows for internal standards and target compounds integrations are updated with each calibration curve and after any instrument maintenance occurs that causes a shift that may affect ChemStation integrations. Retention time windows are set using either method defined limits or by analytical judgment in order to provide the best chance for the software to routinely perform a proper automated integration for the compound. Compounds are identified when the following criteria are met.
- 8.6.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.
- 8.6.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- 8.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
(EXAMPLE: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.) Analyst experience is vital in this determination when interferences are present.
- 8.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

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8.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

8.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

8.7 TICs – Tentatively Identified Compounds

Periodically, clients may request the tentative identification of compounds present in the field sample that are not normal target compounds and are not normally calibrated. This identification is limited to the compounds in the current NBS (National Bureau of Standards) mass spectral library employed by ESC.

Library Search Identification – For samples containing components not associated with the calibration standards, a library search may be made for the purpose of a tentative identification. Data system library searches must not use normalization routines that would misrepresent the library or unknown spectra when making comparisons. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. The analyst may only assign tentative identifications after visual comparison of sample spectra with the nearest library searches.

Guidelines for tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within $\pm 20\%$. (EXAMPLE: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.

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- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Routinely, ESC employs a minimum Q value of 60 for tentative identifications and a minimum concentration of 10ppb. Peaks below those criteria could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology.

8.8 Quantitative analysis

- 8.8.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.
 - 8.8.1.1 It is recommended to use the integrations produced by the software if the integration is correct because the software will produce more consistent integrations of peaks in chromatograms. Manual integrations may be necessary in some cases and must be performed in conjunction with ESC SOP #030215, *Manual Integration*.
- 8.8.2 If the RSD of a compound's response factor meets method requirements, then the concentration in the extract may be determined using the average response factor (average RF) from initial calibration data.
- 8.8.3 Where applicable, the concentration of any tentatively identified compounds in the sample should be estimated. The same formula as is used to calculate target analyte concentrations is used with the following modifications: The areas A_x and A_{is} must be from the total ion chromatograms and the RF for the compound is assumed at 1. See section 9.7 for calculation.
- 8.8.4 The resulting concentration must be reported indicating that the value is an estimate. Use the nearest internal standard free of interferences.

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- 8.8.5 Quantitation of multi-component compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081 or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10ng/ μ L in the concentrated sample extract.
- 8.8.6 **Peak Resolution:** Structural isomers that produce very similar spectra must be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomers is <50% of the average of the two peak heights (for Method 8270D) and <25% of the sum of the two peak heights (for Methods 8270C & 625). Otherwise, structural isomers should be identified as isomeric pairs.

STATE NOTE: Minnesota MPCA requires that peak resolution of all co-eluters, analyzed using Method 8270C, must be resolved as close to 75% as possible. Resolution must be adequate at lower levels and not worsen as concentration increases.

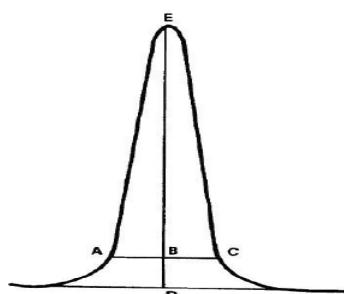
9.0 DATA ANALYSIS AND CALCULATIONS

9.1 GC/MS Tune: DDT Breakdown Determination during Tuning:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

9.2 GC/MS Tune: Benzidine and Pentachlorophenol Tailing Factor

$$\text{TailingFactor} = \frac{BC}{AB}$$



where: BC is the width of the back $\frac{1}{2}$ of the peak at 10% of the peak height
AB is the width of the front $\frac{1}{2}$ of the peak at 10% of the peak height.

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9.3 Internal Calibration Equations (Response Factors):

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate, in µg/L.

C_{is} = Concentration of the internal standard, in µg/L.

- Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100\%$$

where:

RSD = Relative standard deviation.

RF = Mean of 5 initial RFs for a compound.

SD = Standard deviation of average RFs for a compound.

- Concentration of an analyte in an extract using RF:

$$\text{Conc.}_{\text{analyte}} = \frac{(\text{Conc.}_{\text{Std}})(\text{Area}_{\text{Analyte}})}{(\text{Average RF}_{\text{analyte}})(\text{Area}_{\text{Std}})}$$

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9.4 Linear calibration model:

$$y = mx + b$$

where:
y = Response A_X for External Standard
x = Concentration C_X for External Standard
m = Slope
b = Intercept

- Slope (m):

$$m = \frac{[(Swx_i y_i * Sw) - (Swx_i * Swy_i)]}{[(Sw * Swx_i^2) - (Swx_i * Swx_i)]}$$

- Intercept (b):

$$b = y_{AVE} - (m * (x_{AVE}))$$

- Correlation Coefficient (r):

$$r = \frac{[(Sw * Swx_i y_i) - (Swx_i * Swy_i)]}{\sqrt{[(Sw * Swx_i^2) - (Swx_i * Swx_i)] * [(Sw * Swy_i^2) - (Swy_i * Swy_i)]}}$$

- Coefficient of Determination (r^2):

$$r^2 = r * r$$

Where:
n = number of x, y pairs
 x_i = individual values for the independent variable
 y_i = individual values for the dependent variable
w = weighting factor, for equal or no weighting w = 1
 x_{AVE} = average of the x values
 y_{AVE} = average of the y values
S = the sum of all the individual values

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9.5 The quadratic calibration fit is performed using the following equation:

$$y = ax^2 + bx + c$$

where: y = Response A_x for External Standard or A_x/A_{is} for Internal Standard
 x = Concentration C_x for External Standard or C_x/C_{is} for Internal Standard

- Coefficients (a,b,c)

$$a = \frac{\{[S(x^2y) * S(xx)] - [S(xy) * S(xx^2)]\}}{\{[(S(xx) * S(x^2x^2)) - [S(xx^2)]^2\}}$$

$$b = \frac{\{[S(xy) * S(x^2x^2)] - [S(x^2y) * S(xx^2)]\}}{\{[(S(xx) * S(x^2x^2)) - [S(xx^2)]^2\}}$$

$$c = [(Sy_i)/n] - \{b * [(Sx_i)/n]\} - \{a * [S(x_i^2)/n]\}$$

where: $S(xx) = (Sx_i^2) - [(Sx_i)^2/n]$
 $S(xy) = (Sx_iy_i) - [(Sx_i)*(Sy_i)/n]$
 $S(xx^2) = (Sx_i^3) - [(Sx_i)*(Sx_i^2)/n]$
 $S(x^2y) = (Sx_i^2y_i) - [(Sx_i^2)*(Sy_i)/n]$
 $S(x^2x^2) = (Sx_i^4) - [(Sx_i^2)^2/n]$

- Coefficient of Determination (r^2)

$$r^2 = \frac{[S(y_i - y_{AVE})^2] - \{[(n-1) / (n-p)] * [S(y_i - Y_i)^2]\}}{S(y_i - y_{AVE})^2}$$

where: y_i = individual values for each dependent variable
 x_i = individual values for each independent variable
 y_{AVE} = average of the y values
 n = number of pairs of data
 p = number of parameters in the polynomial equation (i.e., 3 for third order, 2 for second order)
 $Y_i = \{(2a*(C_x/C_{is})^2) - b^2 + b + (4ac)\}/(4a)$
 S = the sum of all the individual values

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- Quadratic Regression Equations for Analyte Concentration:

$$C_x = \frac{-b \pm \sqrt{(b^2 - 4ac)} - A_x}{2a}$$

9.6 Continuing Calibration:

- Percent Difference:

$$\% \text{ Difference} = \frac{RF_v - \overline{RF}}{\overline{RF}} \times 100$$

- Percent Drift:

$$\% \text{ Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

9.7 Relative Retention Time (RRT):

$$RRT = \frac{\text{Retention Time of the Analyte}}{\text{Retention Time of the ISTD}}$$

9.8 To calculate the concentration of the analyte in the original samples:

- Water:

$$(\text{Concentration of analyte extract}) \times \frac{(\text{volume of extract})}{(\text{volume of sample})}$$

- Soil:

$$(\text{Concentration of analyte extract}) \times \frac{(\text{volume of extract})}{(\text{weight of sample})}$$

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9.9 Percent Dry Weight

$$\% \text{DryWeight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

9.10 LCS/ICV/CCV Percent Recovery (%R):

$$\% R = \frac{\text{Measured concentration}}{\text{Actual concentration}} \times 100$$

9.11 Matrix Spike Recoveries (%R_{MS/MSD}):

$$\% R_{\text{MS/MSD}} = \frac{O_i - O_s}{T_i} \times 100$$

where: O_i = observed sample concentration with the spike added
 O_s = the observed value for the sample without the spike
 T_i = True value of the spike added

9.12 Relative Percent Difference (%RPD):

$$\text{RPD} = \frac{\text{Value 1} - \text{Value 2}}{\left(\frac{\text{Value 1} + \text{Value 2}}{2} \right)} \times 100$$

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- 9.13 External Standard Calibration (see section 13.1): The calibration factor for each standard can be calculated:

$$CF = \frac{A_s}{C_s}$$

where: A_s - Average Peak Area over the number of peaks used for quantitation
 C_s – Concentration of the analyte in the standard.

- The average (or mean) calibration factor (\overline{CF}) is calculated:

$$\overline{CF} = \frac{\sum_{i=1}^n CF_i}{n}$$

where: CF_i – Calibration Factor for each level of the calibration curve
 n – number of standards analyzed in the calibration curve

- The standard deviation (SD) of the calibration is determined:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n-1}}$$

where: \overline{CF} – Average Calibration Factor for the calibration curve
 CF_i – Calibration Factor for each level of the calibration curve
 n – number of standards analyzed in the calibration curve

- The Percent Relative Standard Deviation for each analyte in the curve is determined:

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

where: SD – Standard Deviation for each analyte
 \overline{CF} – Average calibration factor for the specific analyte

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- Percent Difference for daily calibration curve verification:

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

where: CF_v – Calibration Factor from the calibration verification standard
 \overline{CF} – Average (or mean) calibration factor from the initial calibration curve

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP 030201, *Data Handling and Reporting*.

10.3 Batches:

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 Instrument Tune, 1 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Second Source Verification (SSCV), 1 Matrix Spike/Spike Duplicate (MS/MSD). All batch information must be maintained in the preparation documentation assigned to the department.

10.4 For acceptance criteria for calibration standards, QC samples and field samples and corrective actions, see section 11.0.

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11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 A successful DFTPP tune must be achieved prior to initial calibration or daily calibration verification. If a tune does not meet the acceptance criteria in section 8.2, then re-inject the tuning solution. If the failure persists, instrument maintenance or detector adjustment is required. The instrument is equipped with detector adjustments in routines called "Autotunes" that can make minor adjustments to m/z ratios and detector setting and can align the analytical system to return the system to peak performance. If after performing the Autotune routine, the injected tuning standard still fails, the system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer.

Following successful tuning of the DFTPP solution, the DDT degradation and Benzidine/Pentachlorophenol tailing must be assessed. If either fail to meet the required acceptance criteria, instrument maintenance is required. The DDT degradation is most likely an inlet or column condition and corrective action entails clipping 6-12" from the injector end of the column, changing the injection port liner, possibly changing the gold inlet seal and re-injecting the tuning solution. The tailing issue is most likely caused by the same type of inlet issues and the same corrective action steps should occur when the tailing criteria is not met. Tailing may also be caused by incorrect column positioning in the inlet and the correct position of the column should be verified prior to performing more involved corrective action processes.

A successful instrument tune, including degradation and tailing acceptability, must be achieved prior to the analysis of calibration standards and sample extracts.

11.2 Initial or Continuing Calibration:

Method 8270C, SM 6410B & Method 625: If the calibration curve or daily calibration verification fails to meet the requirements of the initial calibration requirements, samples may not be quantitated using the calibration curve and a new calibration curve must be analyzed. Instrument maintenance or new standard preparation may be required. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer.

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Method 8270D: Due to the large number of compounds that may be analyzed by this method, some compounds in the initial and/or daily calibration verification will fail to meet the initial and continuing calibration acceptance criteria. For these instances, failing compounds may not be critical to specific project needs and therefore may be utilized as qualified data or estimated values for screening purposes. If more than 10% of the compounds in the initial or continuing calibration exceed the 20% RSD limit and/or do not meet the minimum correlation coefficient (0.990) for alternate curve fits, then the chromatographic system is considered too reactive for analysis. Instrument maintenance must be performed and the calibration process must be repeated. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer.

- 11.3 The method blank must be extracted and analyzed with each set of samples and must be free of the analytes of interest at $\frac{1}{2}$ the level being reported (RL). If the method blank contains target analytes at a concentration $>\frac{1}{2}$ the RL, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts. Following verification that the analytical system is free from interferences, the method blank can be re-analyzed once. If the method blank continues to demonstrate target analytes above the $\frac{1}{2}$ criteria, then corrective action is required. The surrogate recoveries in the method blank must meet the established control criteria, listed in Attachments IVa & IVb. If not, the recovery demonstrates an analytical system that is in an out-of-control mode and the batch must be re-extracted unless directed otherwise by the client.

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, the method blank concentration must be less than the reporting limit.

- 11.4 **Method 8270D:** The value determined from the second source calibration verification (SSCV) should be within $\pm 30\%$ of the expected concentration. Alternative recovery limits may be appropriate based on analyte performance and project specific requirements. Quantitative analysis cannot proceed for analytes that fail the SSCV, except for screening purposes only.

Method 8270C/625/SM6410B: The value determined from the second source calibration verification (SSCV) must be $\leq 50\%$ for non-CCC compounds; $\leq 20\%$ for CCC compounds and meet the minimum response factor criteria for SPCC compounds as in the initial calibration construction. Historical performance weighs heavily in the acceptability of those analytes that are known to perform poorly.

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- 11.5 Surrogates: If the surrogate recoveries in the samples do not fall within the established limits (listed in Attachments IVa & IVb), ensure that there were no errors in calculations, internal standard, or instrument performance. If no errors were found, re-extract the sample, unless directed otherwise by the client. If there is no more sample available or it has exceeded holding times, the data must be flagged with a "J1" (surrogate high) or a "J2" (surrogate low). See SOP #030201, *Data Handling and Reporting*, for more information on qualifying out of control data.

STATE NOTE: If the sample is from North Carolina, two of the three acid and two of the three base/neutral surrogates must pass. If two of the three acid or base/neutral surrogates fail, the sample must be re-extracted. For all other samples, one of the three surrogates must pass from both the acid and base/neutral sides. If any surrogates have less than a 10% recovery, the sample must be re-extracted.

STATE NOTE: If field samples are analyzed in conjunction with the Ohio VAP program, surrogate outliers in batch QC samples, including the method blank, LCS/LCSD, MS/MSD require re-extraction of the entire batch, if sufficient volume has been submitted by the client and an obvious matrix interferent is not present.

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. method blank, surrogate, spike, ISTD, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.

- 11.6 Internal Standard: The internal standard area counts must be monitored for all CCVs. ISTDs must recover within -50% to +100% of the area counts from the internal standard area counts of the midpoint standard of the most recent initial calibration sequence. If any internal standard response is beyond the acceptable recovery, corrective action is required. Corrective action can take the form of checking the original calculations to ensure accuracy, re-analysis of the CCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

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Internal standards must be monitored for each sample. ISTDs in samples must meet the -50% to +100% criteria when compared to the ISTDs in the daily CCV or mid-level of the calibration curve, on 12h shifts when full calibration is performed. Possible corrective actions include: if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than +100%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed to confirm the original results.

- 11.7 LCS/LCSD and MS/MSD: The laboratory control sample, laboratory control sample duplicate, matrix spike and matrix spike duplicate recoveries must be evaluated against the limits listed in Attachments IVa & IVb. The LCS/LCSD and MS/MSD are spiked with the same list of compounds for which the instrument is calibrated. Due to the large number of compounds analyzed using these methods, it is statistically likely that accuracy and precision failures will occur.

LCS or LCSD samples that do not pass the acceptable QC criteria must be re-analyzed. LCS/LCSD failures must meet the marginal exceedence criteria below. The normal compound list for 8270/625 typically contains 90 analytes; therefore only 5 analytes can be considered as marginally exceeding the acceptance criteria. If more than 5 failures occur or if the failures demonstrate a pattern that is causing the outliers, the entire sample batch with associated QC must be re-extracted and re-analyzed. Marginal exceedences must be random events.

Upper and lower marginal exceedence (ME) limits are established by +/- four times the standard deviation of historical accuracy data and the number of marginal excedences allowed is based on the number of analytes spiked in the LCS.

Number of allowable marginal excedences:

90 analytes, 5 analytes allowed in the ME limit
71 – 90 analytes, 4 analytes allowed in the ME limit.
51 – 70 analytes, 3 analytes allowed in the ME limit.
31 – 50 analytes, 2 analytes allowed in the ME limit.
11 – 30 analytes, 1 analyte allowed in the ME limit.
< 11 analytes, no analyte allowed in the ME limit.

If the MS/MSD fails to meet recovery limits listed in Attachment II, the data on the unspiked field sample for that compound must be flagged with a "J5" (high recovery) or a "J6" (low recovery). If the MS/MSD fail to pass precision limits, the data on the unspiked field sample for that compound must be flagged with a "J3" qualifier.

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STATE NOTE: For South Carolina and Ohio VAP samples, marginal excedences do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be within 70-130% for both SC matrices with an RPD of 20%.

11.8 Calibration Range: For any compound found in a sample at a level above the highest standard, the extract must be diluted and re-analyzed to allow quantitation within the range of instrument calibration. Whenever an extract dilution is made, the appropriate amount of internal standard must be added to bring the ISTD concentrations back to the original amount added prior to the dilution.

11.8.1 In some cases, where allowed by the client, data exceeding the highest level of calibration can be reported to the client accompanied by an "E" flag. This is only permissible when the client is willing to accept the estimated value. A "No E" list is maintained by the laboratory that includes all clients that do not accept the estimated qualifier.

STATE NOTE: Samples analyzed in conjunction with the states of Arizona, Minnesota, New York, South Carolina, Ohio VAP, or the state Laboratory of Tennessee will not accept estimated values on final reports.

11.9 Second Analyst Data Review: A second analyst who is knowledgeable regarding these methods of analysis reviews all data before results are report. The reviewing process includes checking all times, including tuning, verifying initial and continuing calibrations, verifying surrogate recoveries, internal standard area counts, matrix spike, matrix spike duplicate, laboratory control sample and laboratory control duplicate recoveries. Reviewing must also include checking all dilutions, concentrations, units, any manual calculations and any manual integrations. All flags and methodologies must be checked. See SOP #030201, *Data Handling and Reporting*, and SOP #030227, *Data Review*, for information on second analyst review.

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STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within $\pm 40\%$ of the expected concentration. If this criteria is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

- 11.10 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
 - 11.10.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a "B3" flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blank, the data may be reported with a "B" flag.
 - STATE NOTE:** The Ohio VAP program does not accept data release using the 10X criteria for method blank contamination as noted in section 11.10.1.
 - 11.10.2 If the MS/MSD fails (recovery less than 30% or greater than 150% and/or RPD greater than 30%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.
 - 11.10.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis. This does not apply when MDL/RL reporting is required by the client.
 - 11.10.4 If a duplicate is above the acceptable range for the RPD and the sample concentration is $<5X$ the RL, then the value can be flagged with a "P1" qualifier indicating that the RPD calculation is not applicable at that concentration.

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12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan*.

- 12.2 See SOP #030302, *Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

- 13.1 The **Missouri Department of Natural Resources** requires that DRO be analyzed by GC/MS. Tuning and frequency requirements are the same as 8270C, omitting DDT, pentachlorophenol, and benzidine assessments. Extract samples the same as 8270PAH using the appropriate extraction method. Only base/neutral surrogates are needed. GC/MS mass range should be 35-550amu. Prepare a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 10,000 µg/mL each in methylene chloride. Calibration standards range from 200 to 10,000µg/mL. Retention time windows are set using C₁₀, C₂₁, and C₃₅. For DRO, set RT 0.1 minutes after C₁₀ to 0.1 minutes after C₂₁. For ORO, set RT 0.1 minutes after C₂₁ to 0.1 minutes after C₃₅. Verify RT windows daily (24 hours) by running component standard. Quantitate using baseline-to baseline, not valley-to-valley. The total ion chromatogram must be used to quantitate. DRO is quantitated using external standard method. The response factor determined for DRO (C₁₀-C₂₁) **must** be used for C₂₁-C₃₅. Subtract area for any internal standard and surrogates. %RSD <20. Run a CCV every 12 hours near mid-point of calibration, %D <20. Run a method blank, LCS and MS/MSD every extraction batch. May re-process files to quantitate PAH analytes, if needed. Quantitation of DRO must be performed using the external standard process.
- 13.2 EPA method 625 employs the use of 2 separate packed GC columns for base/neutral and acidic analyte separations. Modern capillary column technology employs a single column that provides sufficient separatory abilities for use in this analytical process as is demonstrated in EPA method 8270C.
- 13.3 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.
- 13.4 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

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14.0 REFERENCES

- 14.1 SW846, Method 8270C, *SVOC by GC/MS*, Rev. 3, December 1996.
- 14.2 SW846, Method 8270D, *SVOC by GC/MS*, Rev. 4, February 2007.
- 14.3 SW846, Method 8000B, *Determinative Chromatographic Separations*, Rev. 2, December 1996.
- 14.4 SW846, Method 8000C, *Determinative Chromatographic Separations*, Rev. 3, March 2003.
- 14.5 40 CFR Part 136, EPA Method 625, October 1991.
- 14.6 Standard Methods for the Examination of Water and Wastewater, APHA, 20th edition, *Method 6410B*.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
13	11/23/10	Technical and Quality Review and update. Revised sections 2.1, 2.10, 4.2 through 4.6, 7.1, 7.6, 7.8, 7.10.2, 7.12, 7.13, 8.3, 8.6, 9.3, 9.4, 9.5, 9.10 through 9.13, 11.3, 11.6, 12.1; Added sections 2.27 through 2.30, 4.7, 7.14, state note following section 11.9, 11.10, and 13.4; Removed section 1.2.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	4/27/95	Origination
1	7/13/95	
2	8/22/96	
3	8/20/99	
4	4/18/00	
5	8/21/00	
6	12/20/00	
7	9/3/01	
8	7/30/02	
9	7/9/03	
10	3/25/04	
11	8/7/06	Technical and Quality Review and update.
12	2/11/09	Addition of 8270D requirements; Addition of State Notes; Update of standards information; Technical and Quality Review and update. Ohio VAP approval 2/11/09.

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Attachment II: 8270/625 Common Calibration List & Reporting Limits (may be updated without notice)

Analyte	Water mg/L	Soil mg/Kg
Acenaphthene	0.001	0.033
Acenaphthylene	0.001	0.033
Acetophenone	0.01	0.33
Anthracene	0.001	0.033
Atrazine	0.01	0.33
Benzaldehyde	0.01	0.33
Benzidine	0.05	0.33
Benzo(a)anthracene	0.001	0.033
Benzo(b)fluoranthene	0.001	0.033
Benzo(k)fluoranthene	0.001	0.033
Benzo(g,h,i)perylene	0.001	0.033
Benzo(a)pyrene	0.001	0.033
Bis(2-chlorethoxy)methane	0.01	0.33
Bis(2-chloroethyl)ether	0.01	0.33
Bis(2-chloroisopropyl)ether	0.01	0.33
4-Bromophenyl-phenylether	0.01	0.33
Caprolactam	0.01	0.33
2-Chloronaphthalene	0.01	0.33
4-Chlorophenyl-phenylether	0.01	0.33
Chrysene	0.001	0.033
Dibenz(a,h)anthracene	0.001	0.033
3,3-Dichlorobenzidine	0.01	0.33
2,4-Dinitrotoluene	0.01	0.33
2,6-Dinitrotoluene	0.01	0.33
Fluoranthene	0.001	0.033
Fluorene	0.001	0.033
Hexachlorobenzene	0.01	0.33
Hexachloro-1,3-butadiene	0.01	0.33
Hexachlorocyclopentadiene	0.01	0.33
Hexachloroethane	0.01	0.33
Indeno(1,2,3-cd)pyrene	0.001	0.033

Analyte	Water mg/L	Soil mg/Kg
Isophorone	0.01	0.33
Naphthalene	0.001	0.033
Nitrobenzene	0.01	0.33
n-Nitrosodimethylamine	0.01	0.33
n-Nitrosodiphenylamine	0.01	0.33
n-Nitrosodi-n-propylamine	0.01	0.33
Phenanthrene	0.001	0.033
Benzylbutyl phthalate	0.001	0.033
Bis(2-ethylhexyl)phthalate	0.001	0.033
Di-n-butyl phthalate	0.001	0.033
Diethyl phthalate	0.001	0.033
Dimethyl phthalate	0.001	0.033
Di-n-octyl phthalate	0.001	0.033
Pyrene	0.001	0.033
1,2,4-Trichlorobenzene	0.01	0.33
4-Chloro-3-methylphenol	0.01	0.33
2-Chlorophenol	0.01	0.33
2,4-Dichlorophenol	0.01	0.33
2,4-Dimethylphenol	0.01	0.33
4,6-Dinitro-2-methylphenol	0.01	0.33
2,4-Dinitrophenol	0.01	0.33
2-Methylphenol	0.01	0.33
4-Methylphenol	0.01	0.33
2-Nitrophenol	0.01	0.33
4-Nitrophenol	0.01	0.33
Pentachlorophenol	0.01	0.33
Phenol	0.01	0.33
2,4,6-Trichlorophenol	0.01	0.33
1-Methylnaphthalene	0.001	0.033
2-Methylnaphthalene	0.001	0.033
4-Chloroaniline	0.01	0.33

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Analyte	Water mg/L	Soil mg/Kg
2-Nitroaniline	0.01	0.33
3-Nitroaniline	0.01	0.33
4-Nitroaniline	0.01	0.33
1,2,3,4-Tetrachlorobenzene	0.05	1.65
1,2,3,5-Tetrachlorobenzene	0.05	1.65
1,2,4,5-Tetrachlorobenzene	0.05	1.65
1,2,4,5-Tetrachlorobenzene	0.05	1.65
1,2-diphenylhydrazine	0.01	0.33
1,3-Dinitrobenzene	0.05	1.65
1,4-Naphthoquinone	0.05	1.65
1-Chloronaphthalene	0.05	1.65
1-Naphthylamine	0.05	1.65
2,3,4,6-Tetrachlorophenol	0.05	1.65
2,3-Dichloroaniline	0.01	0.33
2,6-Dichlorophenol	0.05	1.65
2-Acetylaminofluorene	0.05	1.65
2-Naphthylamine	0.05	1.65
2-Picoline	0.05	1.65
3,3'-Dimethylbenzidine	0.05	1.65
3-Methylcholanthrene	0.05	1.65
4-Aminobiphenyl	0.05	1.65
4-Nitroquinoline-1-oxide	0.05	1.65
5-Nitro-o-toluidine	0.05	1.65
7,12-Dimethylbenz(a)anthracene	0.05	1.65
7H-Dibenzo (c,g) carbazole	0.05	1.65
a,a-Dimethylphenethylamine	0.05	1.65
Acetophenone	0.01	0.33
Alpha-terpineol	0.01	0.33
Aniline	0.01	0.33
Aramite	0.05	1.65
Benzal Chloride	0.05	1.65
Benzo (j) fluoranthene	0.05	1.65

Analyte	Water mg/L	Soil mg/Kg
Benzotrichloride	0.05	1.65
Benzyl Chloride	0.05	1.65
Chlorobenzilate	0.05	1.65
Diallate (cis)	0.05	1.65
Diallate (trans)	0.05	1.65
Dibenz (a,e) pyrene	0.05	1.65
Dibenz (a,h) acridine	0.05	1.65
Dibenz (a,h) pyrene	0.05	1.65
Dibenz (a,i) pyrene	0.05	1.65
Dimethoate	0.05	1.65
Dinoseb	0.05	1.65
Diphenylamine	0.05	1.65
Disulfoton	0.05	1.65
Ethyl methanesulfonate	0.05	1.65
Famphur	0.05	1.65
Hexachlorophene	0.05	1.65
Hexachloropropene	0.05	1.65
Isodrin	0.05	1.65
Isosafrole (cis)	0.05	1.65
Isosafrole (trans)	0.05	1.65
Kepone	0.05	1.65
Methapyrilene	0.05	1.65
Methyl methanesulfonate	0.05	1.65
Methyl parathion	0.05	1.65
N-Nitrosodiethylamine	0.05	1.65
n-nitrosodi-n-butylamine	0.01	0.33
N-Nitrosodi-n-butylamine	0.05	1.65
N-Nitrosomethylethylamine	0.05	1.65
N-Nitrosomorpholine	0.05	1.65
N-Nitrosopiperidine	0.05	1.65
N-Nitrosopyrrolidine	0.05	1.65
o,o,o-Triethylphosphorothioate	0.05	1.65

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Analyte	Water mg/L	Soil mg/Kg
o-cresol	0.01	0.33
o-Toluidine	0.05	1.65
Parathion	0.05	1.65
p-cresol	0.01	0.33
p-Dimethylaminoazobenzene	0.05	1.65
Pentachlorobenzene	0.05	1.65
Pentachloroethane	0.05	1.65
Pentachloronitrobenzene	0.05	1.65
Phenacetin	0.05	1.65
Phorate	0.05	1.65
p-Phenylenamine	0.05	1.65
Pronamide	0.05	1.65
Safrole	0.05	1.65
Sulfotep	0.05	1.65
sym-Trinitrobenzene	0.05	1.65
Thionazin	0.05	1.65
2-nitrodiphenylamine	0.01	0.33
n-decane	0.01	0.33
n-octadecane	0.01	0.33
Pentachlorphenol (SIM)	0.001	-

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Attachment III – Appropriate Extraction Methods by Analyte (printed from SW-846 Method 8270C)

ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Acenaphthene	X	X	X	X	X	83-32-9
Acenaphthene d(10) (IS)	X	X	X	X	X	
Acenaphthylene	X	X	X	X	X	208-96-8
Acetophenone	X	ND	ND	ND	X	98-86-2
2-Acetylaminofluorene	X	ND	ND	ND	X	53-96-3
1-Acetyl-2-thiourea	LR	ND	ND	ND	LR	591-08-2
Aldrin	X	X	X	X	X	309-00-2
2-Aminoanthraquinone	X	ND	ND	ND	X	117-79-3
Aminoazobenzene	X	ND	ND	ND	X	60-09-3
4-Aminobiphenyl	X	ND	ND	ND	X	92-67-1
3-Amino-9-ethylcarbazole	X	X	ND	ND	ND	132-32-1
Anilazine	X	ND	ND	ND	X	101-05-3
Aniline	X	X	ND	X	X	62-53-3
Ortho-anisidine	X	ND	ND	ND	X	90-04-0
Anthracene	X	X	X	X	X	120-12-7
Aramite HS	(43)	ND	ND	ND	X	140-57-8
Aroclor 1016	X	X	X	X	X	12674-11-2
Aroclor 1221	X	X	X	X	X	11104-28-2
Aroclor 1232	X	X	X	X	X	11141-16-5
Aroclor 1242	X	X	X	X	X	53469-21-9
Aroclor 1248	X	X	X	X	X	12672-29-6
Aroclor 1254	X	X	X	X	X	11097-69-1
Aroclor 1260	X	X	X	X	X	11096-82-5
Azinphos-methyl HS	(62)	ND	ND	ND	X	86-50-0
Barban	LR	ND	ND	ND	LR	101-27-9
Benzidine	CP	CP	CP	CP	CP	92-87-5
Benzoic Acid	X	X	ND	X	X	65-85-0
Benz(a)anthracene	X	X	X	X	X	56-55-3
Benzo(b)fluoranthene	X	X	X	X	X	205-99-2
Benzo(k)fluoranthene	X	X	X	X	X	207-08-9
Benzo(g,h,i)perylene	X	X	X	X	X	191-24-2
Benzo(a)pyrene	X	X	X	X	X	50-32-8
Para-benzoquinone	OE	ND	ND	ND	X	106-51-4
Benzyl Alcohol	X	X	ND	X	X	100-51-6
Alpha-BHC	X	X	X	X	X	319-84-6
Beta-BHC	X	X	X	X	X	319-85-7
Delta-BHC	X	X	X	X	X	319-86-8
Gamma-BHC	X	X	X	X	X	58-89-9
Lindane	X	X	X	X	X	58-89-9

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Bis(2-chloroethoxy)methane	X	X	X	X	X	111-91-1
Bis(2-chloroethyl) Ether	X	X	X	X	X	111-44-4
Bis(2-chloroisopropyl) Ether	X	X	X	X	X	108-60-1
Bis(2-ethylhexyl) Phthalate	X	X	X	X	X	117-81-7
4-Bromophenyl Phenyl Ether	X	X	X	X	X	101-55-3
Bromoxynil	X	ND	ND	ND	X	1689-84-5
Butyl Benzyl Phthalate	X	X	X	X	X	85-68-7
Captafol HS	(55)	ND	ND	ND	X	6/1/2425
Captan HS	(40)	ND	ND	ND	X	133-06-2
Carbaryl	X	ND	ND	ND	X	63-25-2
Carbofuran	X	ND	ND	ND	X	1563-66-2
Carbophenothon	X	ND	ND	ND	X	786-19-6
Chlordane	X	X	X	X	X	57-74-9
Chlорfenvinphos	X	ND	ND	ND	X	470-90-6
4-Chloroaniline	X	ND	ND	ND	X	106-47-8
Chlorobenzilate	X	ND	ND	ND	X	510-15-6
5-Chloro-2-methylaniline	X	ND	ND	ND	X	95-79-4
4-Chloro-3-methylphenol	X	X	X	X	X	59-50-7
hydrochloride	X	ND	ND	ND	X	6959-48-4
1-Chloronaphthalene	X	X	X	X	X	90-13-1
2-Chloronaphthalene	X	X	X	X	X	91-58-7
2-Chlorophenol	X	X	X	X	X	95-57-8
4-Chloro-1,2-phenylenediamine	X	X	ND	ND	ND	95-83-0
4-Chloro-1,3-phenylenediamine	X	X	ND	ND	ND	5131-60-2
4-Chlorophenyl Phenyl Ether	X	X	X	X	X	7005-72-3
Chrysene	X	X	X	X	X	218-01-9
Chrysene d(12) (IS)	X	X	X	X	X	
Coumaphos	X	ND	ND	ND	X	56-72-4
Para-cresidine	X	ND	ND	ND	X	120-71-8
Crotoxyphos	X	ND	ND	ND	X	7700-17-6
2-Cyclohexyl-4,6-dinitrophenol	X	ND	ND	ND	LR	131-89-5
4,"-DDD	X	X	X	X	X	72-54-8
4,"-DDE	X	X	X	X	X	72-55-9
4,"-DDT	X	X	X	X	X	50-29-3
Demeton-O HS	(68)	ND	ND	ND	X	298-03-3
Demeton-S	X	ND	ND	ND	X	126-75-0
Diallate (cis or trans)	X	ND	ND	ND	X	2303-16-4
2,4-Diaminotoluene DC,	OE(42)	ND	ND	ND	X	95-80-7
Dibenz(a,j)acridine	X	ND	ND	ND	X	224-42-0

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Dibenz(a,h)anthracene	X	X	X	X	X	53-70-3
Dibenzofuran	X	X	ND	X	X	132-64-9
Dibenzo(a,e)pyrene	ND	ND	ND	ND	X	192-65-4
1,2-Dibromo-3-chloropropane	X	X	ND	ND	ND	96-12-8
Di-n-butyl Phthalate	X	X	X	X	X	84-74-2
Dichlone	OE	ND	ND	ND	X	117-80-6
1,2-Dichlorobenzene	X	X	X	X	X	95-50-1
1,3-Dichlorobenzene	X	X	X	X	X	541-73-1
1,4-Dichlorobenzene	X	X	X	X	X	106-46-7
1,4-Dichlorobenzene d(4)(IS)	X	X	X	X	X	
3,"-Dichlorobenzidine	X	X	X	X	X	91-94-1
2,4-Dichlorophenol	X	X	X	X	X	120-83-2
2,6-Dichlorophenol	X	ND	ND	ND	X	87-65-0
Dichlorovos	X	ND	ND	ND	X	62-73-7
Dicrotophos	X	ND	ND	ND	X	141-66-2
Dieldrin	X	X	X	X	X	60-57-1
Diethyl Phthalate	X	X	X	X	X	84-66-2
Diethylstilbestrol	AW,OS(67)	ND	ND	ND	X	56-53-1
Diethyl Sulfate	LR	ND	ND	ND	LR	64-67-5
Dihydrosaffrole	ND	ND	ND	ND	ND	56312-13-1
Dimethoate	HE,HS	ND	ND	ND	X	60-51-5
3,"-Dimethoxybenzidine	X	ND	ND	ND	LR	119-90-4
Dimethylaminoazobenzene	X	ND	ND	ND	X	60-11-7
7,12-Dimethylbenz(a)-anthracene	CP(45)	ND	ND	ND	CP	57-97-6
3,"-Dimethylbenzidine	X	ND	ND	ND	X	119-93-7
Alpha,alpha-Dimethylphenethylamine	ND	ND	ND	ND	X	122-09-8
2,4-Dimethylphenol	X	X	X	X	X	105-67-9
Dimethyl Phthalate	X	X	X	X	X	131-11-3
1,2-Dinitrobenzene	X	ND	ND	ND	X	528-29-0
1,3-Dinitrobenzene	X	ND	ND	ND	X	99-65-0
1,4-Dinitrobenzene	HE(14)	ND	ND	ND	X	100-25-4
4,6-Dinitro-2-methylphenol	X	X	X	X	X	534-52-1
2,4-Dinitrophenol	X	X	X	X	X	51-28-5
2,4-Dinitrotoluene	X	X	X	X	X	121-14-2
2,6-Dinitrotoluene	X	X	X	X	X	606-20-2
Dinocap	CP,HS(28)	ND	ND	ND	CP	39300-45-3
Dinoseb	X	ND	ND	ND	X	88-85-7
Dioxathion	ND	ND	ND	ND	ND	78-34-2
Diphenylamine	X	X	X	X	X	122-39-4

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
5,5-Diphenylhydantoin	X	ND	ND	ND	X	57-41-0
1,2-Diphenylhydrazine	X	X	X	X	X	122-66-7
Di-n-octyl Phthalate	X	X	X	X	X	117-84-0
Disulfoton	X	ND	ND	ND	X	298-04-4
Endosulfan I	X	X	X	X	X	959-98-8
Endosulfan II	X	X	X	X	X	33212-65-9
Endosulfan Sulfate	X	X	X	X	X	1031-07-8
Endrin	X	X	X	X	X	72-20-8
Endrin Aldehyde	X	X	X	X	X	7421-93-4
Endrin Ketone	X	X	ND	X	X	53494-70-5
EPN	X	ND	ND	ND	X	2104-64-5
Ethion	X	ND	ND	ND	X	563-12-2
Ethyl Carbamate	DC(28)	ND	ND	ND	X	51-79-6
Ethyl Methanesulfonate	X	ND	ND	ND	X	62-50-0
Famphur	X	ND	ND	ND	X	52-85-7
Fensulfothion	X	ND	ND	ND	X	115-90-2
Fenthion	X	ND	ND	ND	X	55-38-9
Fluchloralin	X	ND	ND	ND	X	33245-39-5
Fluoranthene	X	X	X	X	X	206-44-0
Fluorene	X	X	X	X	X	86-73-7
2-Fluorobiphenyl (Surr)	X	X	X	X	X	321-60-8
2-Fluorophenol (Surr)	X	X	X	X	X	367-12-4
Heptachlor	X	X	X	X	X	76-44-8
Heptachlor Epoxide	X	X	X	X	X	1024-57-3
Hexachlorobenzene	X	X	X	X	X	118-74-1
Hexachlorobutadiene	X	X	X	X	X	87-68-3
Hexachlorocyclopentadiene	X	X	X	X	X	77-47-4
Hexachloroethane	X	X	X	X	X	67-72-1
Hexachlorophene	AW,CP(62)	ND	ND	ND	CP	70-30-4
Hexachloropropene	X	ND	ND	ND	X	1888-71-7
Hexamethylphosphoramide	X	ND	ND	ND	X	680-31-9
Hydroquinone	ND	ND	ND	ND	X	123-31-9
Indeno(1,2,3-cd)pyrene	X	X	X	X	X	193-39-5
Isodrin	X	ND	ND	ND	X	465-73-6
Isophorone	X	X	X	X	X	78-59-1
Isosafrole	DC(46)	ND	ND	ND	X	120-58-1
Kepone	X	ND	ND	ND	X	143-50-0
Leptophos	X	ND	ND	ND	X	21609-90-5
Malathion	HS(5)	ND	ND	ND	X	121-75-5

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Maleic Anhydride	HE	ND	ND	ND	X	108-31-6
Mestranol	X	ND	ND	ND	X	72-33-3
Methapyrilene	X	ND	ND	ND	X	91-80-5
Methoxychlor	X	ND	ND	ND	X	72-43-5
3-Methylcholanthrene	X	ND	ND	ND	X	56-49-5
4,"-Methylenebis (2-chloroaniline)	OE,OS(0)	ND	ND	ND	LR	101-14-4
4,"-Methylenebis-(N-n-dimethylaniline)	X	X	ND	ND	ND	101-61-1
Methyl methanesulfonate	X	ND	ND	ND	X	66-27-3
2-Methylnaphthalene	X	X	ND	X	X	91-57-6
Methyl Parathion	X	ND	ND	ND	X	298-00-0
2-Methylphenol	X	ND	ND	ND	X	95-48-7
3-Methylphenol	X	ND	ND	ND	X	108-39-4
4-Methylphenol	X	ND	ND	ND	X	106-44-5
2-Methylpyridine	X	X	ND	ND	ND	109-06-8
Mevinphos	X	ND	ND	ND	X	7786-34-7
Mexacarbate	HE,HS(68)	ND	ND	ND	X	315-18-4
Mirex	X	ND	ND	ND	X	2385-85-5
Monocrotophos	HE	ND	ND	ND	X	6923-22-4
Naled	X	ND	ND	ND	X	300-76-5
Naphthalene	X	X	X	X	X	91-20-3
Naphthalene d(8) (IS)	X	X	X	X	X	
1,4-Naphthoquinone	X	ND	ND	ND	X	130-15-4
1-Naphthylamine	OS(44)	ND	ND	ND	X	134-32-7
2-Naphthylamine	X	ND	ND	ND	X	91-59-8
Nicotine	DE(67)	ND	ND	ND	X	54-11-5
5-Nitroacenaphthene	X	ND	ND	ND	X	602-87-9
2-Nitroaniline	X	X	ND	X	X	88-74-4
3-Nitroaniline	X	X	ND	X	X	99-09-2
4-Nitroaniline	X	X	ND	X	X	100-01-6
5-Nitro-o-anisidine	X	ND	ND	ND	X	99-59-2
Nitrobenzene	X	X	X	X	X	98-95-3
Nitrobenzene-d(5) (Surr)	X	X	X	X	X	
4-Nitrobiphenyl	X	ND	ND	ND	X	92-93-3
Nitrofen	X	ND	ND	ND	X	1836-75-5
2-Nitrophenol	X	X	X	X	X	88-75-5
4-Nitrophenol	X	X	X	X	X	100-02-7
5-Nitro-o-toluidine	X	ND	ND	ND	X	99-55-8
Nitroquinoline-1-oxide	X	ND	ND	ND	X	56-57-5
N-nitrosodi-n-butylamine	X	ND	ND	ND	X	924-16-3

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
N-nitrosodiethylamine	X	ND	ND	ND	X	55-18-5
N-nitrosodimethylamine	X	X	X	X	X	62-75-9
N-nitrosomethylethylamine	X	ND	ND	ND	X	10595-95-6
N-nitrosodiphenylamine	X	X	X	X	X	86-30-6
N-nitrosodi-n-propylamine	X	X	X	X	X	621-64-7
N-nitrosomorpholine	ND	ND	ND	ND	X	59-89-2
N-nitrosopiperidine	X	ND	ND	ND	X	100-75-4
N-nitrosopyrrolidine	X	ND	ND	ND	X	930-55-2
Octamethyl Pyrophosphoramide	LR	ND	ND	ND	LR	152-16-9
Parathion	X	ND	ND	ND	X	56-38-2
Pentachlorobenzene	X	ND	ND	ND	X	608-93-5
Pentachloronitrobenzene	X	ND	ND	ND	X	82-68-8
Pentachlorophenol	X	X	X	X	X	87-86-5
Perylene d(12) (IS)	X	X	X	X	X	
Phenacetin	X	ND	ND	ND	X	62-44-2
Phenanthrene	X	X	X	X	X	85-01-8
Phenanthrene d(10) (IS)	X	X	X	X	X	
Phenobarbital	X	ND	ND	ND	X	50-06-6
Phenol DC(28)	X	X	X	X		108-95-2
Phenol d(6) (Surr)	DC(28)	X	X	X	X	
1,4-Phenylenediamine	X	ND	ND	ND	X	106-50-3
Phorate	X	ND	ND	ND	X	298-02-2
Phosalone	HS(65)	ND	ND	ND	X	2310-17-0
Phosmet	HS(15)	ND	ND	ND	X	732-11-6
Phosphamidon	HE(63)	ND	ND	ND	X	13171-21-6
Phthalic Anhydride	CP,ME(1)	ND	ND	ND	CP	85-44-9
2-Picoline	X	X	ND	ND	ND	109-06-8
Piperonyl Sulfoxide	X	ND	ND	ND	X	120-62-7
Pronamide	X	ND	ND	ND	X	23950-58-5
Pyrene	X	X	X	X	X	129-00-0
Pyridine	ND	ND	ND	ND	ND	110-86-1
Resorcinol	X	ND	ND	ND	X	94-59-7
Safrole	AW,OS(55)	ND	ND	ND	X	60-41-3
Sulfallate	X	ND	ND	ND	X	95-06-7
Terbufos	X	ND	ND	ND	X	13071-79-9
Terphenyl d(l4)(surr)	X	X	ND	X	X	1718-51-0
1,2,4,5-Tetrachlorobenzene	X	ND	ND	ND	X	95-94-3
2,3,4,6-Tetrachlorophenol	X	ND	ND	ND	X	58-90-2
Tetrachlorvinphos	X	ND	ND	ND	X	961-11-5

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Tetraethyl Dithiopyrophosphate	X	X	ND	ND	ND	3689-24-5
Tetraethyl Pyrophosphate	X	ND	ND	ND	X	107-49-3
Thionazine	X	ND	ND	ND	X	297-97-2
Thiophenol	X	ND	ND	ND	X	108-98-5
Benzene-thiol	X	ND	ND	ND	X	108-98-5
Toluene Diisocyanate	HE(6)	ND	ND	ND	X	584-84-9
Ortho-toluidine	X	ND	ND	ND	X	95-53-4
Toxaphene	X	X	X	X	X	8001-35-2
2,4,6-Tribromophenol (Surr)	X	X	X	X	X	
1,2,4-Trichlorobenzene	X	X	X	X	X	120-82-1
2,4,5-Trichlorophenol	X	X	ND	X	X	95-95-4
2,4,6-Trichlorophenol	X	X	X	X	X	88-06-2
Trifluralin	X	ND	ND	ND	X	1582-09-8
2,4,5-Trimethylaniline	X	ND	ND	ND	X	137-17-7
Trimethyl Phosphate	HE(60)	ND	ND	ND	X	512-56-1
1,3,5-Trinitrobenzene	X	ND	ND	ND	X	99-35-4
Tris(2,3-dibromopropyl) phosphate	X	ND	ND	ND	LR	126-72-7
O,O,O-Triethyl Phosphorothioate	X	ND	ND	ND	X	126-68-1

KEY TO ANALYTE LIST ESC extraction technique Chemical Abstract Service Registry Number

(b) See Sec. 1.2 for other acceptable preparation methods.

(IS) This compound may be used as an internal standard.

(surr) This compound may be used as a surrogate.

(AW) Adsorption to walls of glassware during extraction and storage.

(CP) Nonreproducible chromatographic performance.

(DC) Unfavorable distribution coefficient (number in parenthesis is percent recovery).

(HE) Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).

(HS) Hydrolysis during storage (number in parenthesis is percent stability).

(LR) Low response.

(ND) Not determined.

(OE) Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).

(OS) Oxidation during storage (number in parenthesis is percent stability).

(X) Greater than 70 percent recovery by this technique.

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Attachment IVa 8270/625-- Common Calibration List & ESC- (may be updated without notice)

Compound Name	LCS / LCSD Limits				MS/MSD Limits			
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
1,2,4,5-TETRACHLOROBENZENE	39	116	33	26	129	27	126	46
1,2,4-TRICHLOROBENZENE	26	103	38	13	116	18	105	50
1,2-DICHLOROBENZENE	21	108	50	10	122	10	112	50
1,3-DICHLOROBENZENE	18	108	50	10	123	10	110	50
1,4-DICHLOROBENZENE	19	103	50	10	117	10	108	50
1-METHYLNAPHTHALENE	40	108	35	29	120	10	139	40
2,4,5-TRICHLOROPHENOL	48	120	29	36	131	10	136	45
2,4,6-TRICHLOROPHENOL	49	118	28	37	130	10	137	42
2,4-DICHLOROPHENOL	46	115	28	35	126	10	133	50
2,4-DIMETHYLPHENOL	40	124	36	26	138	10	142	36
2,4-DINITROPHENOL	10	125	50	10	146	10	150	50
2,4-DINITROTOLUENE	56	128	24	44	140	32	137	36
2,6-DINITROTOLUENE	56	121	23	45	132	35	123	37
2-CHLORONAPHTHALENE	44	110	30	33	121	33	109	39
2-CHLOROPHENOL	38	114	36	25	127	10	155	50
2-METHYLNAPHTHALENE	28	122	36	12	138	21	125	42
2-METHYLPHENOL	42	99	26	32	109	13	110	23
2-NITROANILINE	55	124	22	43	136	31	135	26
2-NITROPHENOL	35	118	35	21	132	12	121	48
3,3-DICHLOROBENZIDINE	46	145	31	30	161	10	135	40
3-NITROANILINE	39	141	32	21	158	0	139	50
4,6-DINITRO-2-METHYLPHENOL	24	119	50	10	135	0	138	50
4-BROMOPHENYL-PHENYLETHER	45	105	26	35	115	35	102	23
4-CHLORO-3-METHYLPHENOL	47	116	22	35	127	10	136	29
4-CHLOROANILINE	21	151	36	0	172	0	169	50
4-CHLOROPHENYL-PHENYLETHER	49	116	26	38	127	39	116	32
4-NITROANILINE	43	144	34	26	161	0	166	50
4-NITROPHENOL	10	66	37	10	78	13	59	50
ACENAPHTHENE	48	110	26	37	120	39	112	37
ACENAPHTHYLENE	48	113	28	37	124	37	114	35
ACETOPHENONE	35	98	38	24	109	20	103	44
ANILINE	0	159	50	0	190	0	115	50
ANTHRACENE	55	127	24	43	139	44	136	24
ATRAZINE	43	159	26	24	178	12	159	36
AZOBENZENE	50	129	28	37	142	33	133	32
BENZALDEHYDE	1	78	49	0	91	0	65	50
BENZIDINE	0	46	50	0	56	0	25	50
BENZO(A)ANTHRACENE	57	115	20	47	125	43	117	25
BENZO(A)PYRENE	63	125	22	52	136	33	137	34

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Compound Name	LCS / LCSD Limits				MS/MSD Limits				RPD Limit	
	Control Limits		RPD Limit	Marginal Excedence		Control Limits				
	Lower	Upper		Lower	Upper	Lower	Upper			
BENZO(B)FLUORANTHENE	50	123	32	38	135	35	128	50		
BENZO(G,H,I)PERYLENE	39	143	31	21	160	10	139	50		
BENZO(K)FLUORANTHENE	45	126	37	32	140	36	119	40		
BENZOIC ACID	0	76	50	0	91	1	52	50		
BENZYL ALCOHOL	33	104	32	21	116	5	115	50		
BENZYLBUTYL PHTHALATE	22	154	29	10	175	47	121	28		
BIPHENYL	45	111	30	34	122	31	112	40		
BIS(2-CHLORETHOXY)METHANE	42	116	38	30	128	21	135	39		
BIS(2-CHLOROETHYL)ETHER	26	115	50	11	130	10	134	50		
BIS(2-CHLOROISOPROPYL)ETHER	32	115	47	18	129	14	124	40		
BIS(2-ETHYLHEXYL)PHTHALATE	47	143	24	31	159	10	115	33		
CAPROLACTAM	11	33	37	7	37	0	50	50		
CARBAZOLE	49	133	29	35	147	31	145	29		
CHRYSENE	58	113	21	49	123	41	117	24		
DIBENZ(A,H)ANTHRACENE	39	144	30	22	161	10	145	50		
DIBENZOFURAN	50	121	26	38	133	36	127	36		
DIETHYL PHTHALATE	36	128	27	20	144	23	132	35		
DIMETHYL PHTHALATE	10	135	33	10	156	42	107	27		
DI-N-BUTYL PHTHALATE	51	131	22	37	144	46	121	27		
DI-N-OCTYL PHTHALATE	51	138	22	37	152	22	109	31		
FLUORANTHENE	53	119	28	42	131	36	130	27		
FLUORENE	49	116	25	38	127	37	120	30		
HEXACHLORO-1,3-BUTADIENE	21	116	50	10	132	16	118	50		
HEXACHLOROBENZENE	51	121	23	39	133	41	114	28		
HEXACHLOROCYCLOPENTADIENE	4	126	50	0	147	0	132	50		
HEXACHLOROETHANE	15	109	50	10	125	10	125	50		
INDENO(1,2,3-CD)PYRENE	40	143	30	23	160	10	138	50		
ISOPHORONE	48	126	31	35	139	32	131	38		
M&P-CRESOL	18	51	31	13	57	10	56	36		
NAPHTHALENE	29	103	45	17	115	14	114	50		
NITROBENZENE	31	105	43	18	117	14	122	46		
N-NITROSODIMETHYLAMINE	11	69	50	1	79	0	75	50		
N-NITROSODI-N-PROPYLAMINE	47	122	33	34	134	20	145	43		
N-NITROSODIPHENYLAMINE	59	143	23	45	157	10	171	34		
O-CRESOL	42	99	26	32	109	10	111	24		
PENTACHLOROPHENOL	20	122	50	3	140	0	137	50		
PHENANTHRENE	54	112	22	45	122	38	121	26		
PHENOL	17	52	33	11	58	10	68	32		
PYRENE	46	130	28	32	144	27	136	33		
PYRIDINE	7	48	50	1	55	0	70	50		

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Compound Name	LCS / LCSD Limits				MS/MSD Limits			
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
2-NITRODIPHENYLAMINE	50	137	20	36	151	70	130	20
N-DECANE	10	92	20	0	108	70	130	20
N-OCTADECANE	20	141	20	0	161	70	130	20

Surrogates

P-TERPHENYL-D14	34	149
PHENOL-D5	10	67
NITROBENZENE-D5	12	120
2-FLUOROPHENOL	10	87
2-FLUOROBIPHENYL	26	122
2,4,6-TRIBROMOPHENOL	10	148

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be within 70-130% of the expected target concentration with an RPD of <20%.

Attachment IVb 8270/625 - Common Calibration List & ESC-calculated soil limits as of 12/03/07

Compound Name	LCS / LCSD Limits				MS/MSD Limits			
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
1,2,4,5-TETRACHLOROBENZENE	51	112	21	41	123	47	111	20
1,2,4-TRICHLOROBENZENE	46	99	24	37	108	37	104	26
1,2-DICHLOROBENZENE	46	102	25	37	111	35	109	29
1,3-DICHLOROBENZENE	45	102	28	36	111	31	112	27
1,4-DICHLOROBENZENE	43	100	26	33	110	31	107	31
1-METHYLNAPHTHALENE	53	102	21	44	110	50	103	23
2,4,5-TRICHLOROPHENOL	53	110	25	44	120	28	128	29
2,6-DINITROTOLUENE	56	113	22	46	123	53	114	22
2-CHLORONAPHTHALENE	55	103	20	47	111	52	101	20
2-CHLOROPHENOL	52	108	24	42	118	41	112	27
2-METHYLNAPHTHALENE	52	107	21	43	116	48	109	22
2-METHYLPHENOL	58	116	22	48	126	56	111	20
2-NITROANILINE	54	116	24	44	126	52	117	24
2-NITROPHENOL	38	110	24	26	122	23	117	31
3,3-DICHLOROBENZIDINE	24	123	35	7	139	10	133	41
3-NITROANILINE	17	135	33	0	154	5	134	30
4,6-DINITRO-2-METHYLPHENOL	34	111	33	21	124	10	124	38
4-BROMOPHENYL-PHENYLETHER	47	98	23	39	106	37	103	23
4-CHLORO-3-METHYLPHENOL	54	116	23	44	127	52	119	24
4-CHLOROANILINE	18	130	31	0	149	4	134	28
4-CHLOROPHENYL-PHENYLETHER	55	106	22	47	115	53	105	20
4-NITROANILINE	16	133	37	0	152	12	129	34

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Compound Name	LCS / LCSD Limits				MS/MSD Limits				RPD Limit	
	Control Limits		RPD Limit	Marginal Excedence		Control Limits				
	Lower	Upper		Lower	Upper	Lower	Upper			
4-NITROPHENOL	34	123	36	19	138	15	140	40		
ACENAPHTHENE	54	102	20	46	110	52	102	23		
ACENAPHTHYLENE	56	104	20	48	112	54	103	22		
ACETOPHENONE	42	92	22	34	100	38	94	22		
ANILINE	0	157	33	0	186	0	143	31		
ANTHRACENE	57	112	21	48	121	55	114	21		
ATRAZINE	40	143	25	23	160	40	144	21		
AZOBENZENE	55	118	24	45	129	51	118	23		
BENZALDEHYDE	0	69	32	0	83	0	100	37		
BENZIDINE	0	13	50	0	16	0	45	50		
BENZO(A)ANTHRACENE	55	105	21	47	113	37	124	33		
BENZO(A)PYRENE	59	114	22	50	123	44	129	27		
BENZO(B)FLUORANTHENE	44	116	33	32	128	28	135	33		
BENZO(G,H,I)PERYLENE	41	127	29	27	141	25	123	35		
BENZO(K)FLUORANTHENE	36	119	37	22	133	41	116	34		
BENZOIC ACID	0	113	50	0	132	0	115	50		
BENZYL ALCOHOL	53	115	23	43	125	47	112	25		
BENZYLBUTYL PHTHALATE	57	130	27	45	142	45	143	39		
BIPHENYL	54	103	21	45	112	49	103	24		
BIS(2-CHLORETHOXY)METHANE	52	107	21	43	116	48	108	23		
BIS(2-CHLOROETHYL)ETHER	38	115	28	25	127	36	115	30		
BIS(2-CHLOROISOPROPYL)ETHER	49	106	25	40	115	44	109	27		
BIS(2-ETHYLHEXYL)PHTHALATE	50	130	29	36	143	40	128	34		
CAPROLACTAM	43	131	24	28	146	26	140	27		
CARBAZOLE	42	120	26	29	133	43	122	25		
CHRYSENE	54	103	23	46	111	39	119	31		
DIBENZ(A,H)ANTHRACENE	42	128	28	28	142	29	123	30		
DIBENZOFURAN	56	111	21	47	121	54	111	21		
DIETHYL PHTHALATE	57	110	20	48	119	51	113	21		
DIMETHYL PHTHALATE	57	108	20	49	117	54	108	23		
DI-N-BUTYL PHTHALATE	56	121	22	45	132	49	121	22		
DI-N-OCTYL PHTHALATE	50	128	26	37	141	40	132	27		
FLUORANTHENE	51	109	26	42	119	23	143	29		
FLUORENE	53	106	20	45	115	53	107	22		
HEXACHLORO-1,3-BUTADIENE	46	110	25	36	121	39	113	26		
HEXACHLOROBENZENE	51	117	24	40	128	49	108	27		
HEXACHLOROCYCLOPENTADIENE	21	127	40	3	145	10	131	39		
HEXACHLOROETHANE	43	104	27	33	114	25	118	35		
INDENO(1,2,3-CD)PYRENE	42	127	28	28	141	28	125	32		
ISOPHORONE	56	116	21	45	127	51	115	22		

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Compound Name	LCS / LCSD Limits				MS/MSD Limits				RPD Limit	
	Control Limits		RPD Limit	Marginal Excedence		Control Limits				
	Lower	Upper		Lower	Upper	Lower	Upper			
M&P-CRESOL	30	68	29	24	74	25	67	32		
NAPHTHALENE	46	97	23	37	105	41	100	26		
NITROBENZENE	46	102	23	37	111	40	102	24		
N-NITROSODIMETHYLAMINE	35	111	35	22	123	20	116	38		
N-NITROSODI-N-PROPYLAMINE	54	113	21	44	123	54	110	23		
N-NITROSODIPHENYLAMINE	66	126	22	56	136	54	138	26		
O-CRESOL	58	116	22	48	126	56	111	20		
PENTACHLOROPHENOL	37	118	28	24	131	10	146	35		
PHENANTHRENE	56	102	20	48	110	37	125	27		
PHENOL	55	115	22	44	125	52	111	22		
PYRENE	53	111	26	43	121	22	151	38		
PYRIDINE	22	86	41	11	97	10	97	36		
2-NITRODIPHENYLAMINE	61	120	20	51	130	70	130	20		
N-DECANE	39	78	20	32	85	70	130	20		
N-OCTADECANE	51	114	20	41	125	70	130	20		

Surrogates

P-TERPHENYL-D14	23	143
PHENOL-D5	37	141
NITROBENZENE-D5	18	119
2-FLUOROPHENOL	26	130
2-FLUOROBIPHENYL	30	120
2,4,6-TRIBROMOPHENOL	25	137

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be within 70-130% of the expected target concentration with an RPD of <20%.

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Attachment V: Characteristic Masses (m/z) for Extractable Organic Compounds
(Reprinted from SW-846 Method 8270C /Dec. 1996)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Pyridine	79	52,78,51
N-Nitrosodimethylamine	42	74,44
2-Picoline	93	66,92
Aniline	93	66,65
Phenol	94	65,66
Benzaldehyde	105	106,77,51
Bis(2-chloroethyl) ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene-d4 (IS)	152	150,115
1,4-Dichlorobenzene	146	148,111
Benzyl alcohol	108	79,77
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Bis(2-chloroisopropyl) ether	45	77,121
Methyl methanesulfonate	80	79,65,95
N-Nitrosodi-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
N-Nitrosodiethylamine	102	42,57,44,56
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	122	107,121
Bis(2-chloroethoxy)methane	93	95,123
Benzoic acid	122	105,77
2,4-Dichlorophenol	162	164,98
Ethyl methanesulfonate	79	109,97,45,65
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d8 (IS)	136	68
Naphthalene	128	129,127
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56,42
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
2-Methylphenol	107	108,77,79,90

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Hexachloropropene	213	211,215,117,106,141
Hexachlorocyclopentadiene	237	235,272
N-Nitrosopyrrolidine	100	41,42,68,69
Acetophenone	105	71,51,120
4-Methylphenol	107	108,77,79,90
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
o-Toluidine	106	107,77,51,79
3-Methylphenol	107	108,77,79,90
2-Chloronaphthalene	162	127,164
N-Nitrosopiperidine	114	42,55,56,41
1-Chloronaphthalene	162	127,164
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
2,6-Dinitrotoluene	165	63,89
3-Nitroaniline	138	108,92
Acenaphthene-d10 (IS)	164	162,160
Acenaphthene	154	153,152
2,4-Dinitrophenol	184	63,154
2,6-Dinitrophenol	162	164,126,98,63
4-Chloroaniline	127	129,65,92
Isosafrole	162	131,104,77,51
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
4-Nitrophenol	139	109,65
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	104,102,76,50,130
Diethyl phthalate	149	177,150
Fluorene	166	165,167
N-Nitrosodi-n-butylamine	84	57,41,116,158
4-Chlorophenyl phenyl ether	204	206,141
Atrazine	200	215,58
4,6-Dinitro-2-methylphenol	198	51,105
N-Nitrosodiphenylamine	169	168,167
Safrole	162	104,77,103,135
Diphenylamine	169	168,167
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1-Naphthylamine	143	115,89,63
4-Bromophenyl phenyl ether	248	250,141
2,4,5-Trichlorophenol	196	198,97,132,99
Hexachlorobenzene	284	142,249
Pentachlorophenol	266	264,268
5-Nitro-o-toluidine	152	77,79,106,94
Thionazine	107	96,97,143,79,68
4-Nitroaniline	138	65,108,92,80,39
Phenanthrene-d10 (IS)	188	94,80
Phenanthrene	178	179,176
Anthracene	178	176,179
Carbazole	167	166,168,139
1,3-Dinitrobenzene	168	76,50,75,92,122
Diallate (cis or trans)	86	234,43,70
Pentachlorobenzene	250	252,108,248,215,254
Pentachloronitrobenzene	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	174	101,128,75,116
Di-n-butyl phthalate	149	150,104
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
Demeton-O	88	89,60,61,115,171
Fluoranthene	202	101,203
1,3,5-Trinitrobenzene	75	74,213,120,91,63
Benzidine	184	92,185
Pyrene	202	200,203
Phorate	75	121,97,93,260
Demeton-S	88	60,81,89,114,115
Phenacetin	108	180,179,109,137,80
Dimethoate	87	93,125,143,229
4-Aminobiphenyl	169	168,170,115
, -Dimethylphenylamine	58	91,65,134,42
Pronamide	173	175,145,109,147
Dinoseb	211	163,147,117,240
Disulfoton	88	97,89,142,186
Butyl benzyl phthalate	149	91,206
Methyl parathion	109	125,263,79,93
Dimethylaminoazobenzene	225	120,77,105,148,42
Benz(a)anthracene	228	229,226

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Chrysene-d12 (IS)	240	120,236
3,3'-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
Kepone	272	274,237,178,143,270
Parathion	109	97,291,139,155
Bis(2-ethylhexyl) phthalate	149	167,279
3,3'-Dimethylbenzidine	212	106,196,180
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
Di-n-octyl phthalate	149	167,43
Aramite	185	191,319,334,197,321
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Famphur	218	125,93,109,217
Benzo(a)pyrene	252	253,125
Perylene-d12 (IS)	264	260,265
7,12-Dimethylbenz(a)anthracene	256	241,239,120
2-Acetylaminofluorene	181	180,223,152
3-Methylcholanthrene	268	252,253,126,134,113
Dibenz(a,j)acridine	279	280,277,250
Indeno(1,2,3-cd)pyrene	276	138,227
Dibenz(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
Hexachlorophene	196	198,209,211,406,408
1,2-Diphenylhydrazine/Azobenzene	77	105,182
Surrogates		
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64
Nitrobenzene-d5 (surr)	82	128,54
Phenol-d6 (surr)	99	42,71
Terphenyl-d14 (surr)	244	122,212
2,4,6-Tribromophenol (surr)	330	332,141

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Attachment VI - QC Acceptance Criteria for Method 625

Compound	Test conc. (μ g/L)	Limit for s (μ g/L)	Range for x (μ g/L)	Range p, p(s) (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39	7.2-152.2	D-166
Anthracene	100	32	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
beta-BHC	100	31.5	41.5-130.6	24-149
delta-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23	64.9-114.4	53-127
2-Chloronaphthalene	100	13	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31	D-134.5	D-145
4,4'-DDE	100	32	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137

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Compound	Test conc. ($\mu\text{g}/\text{L}$)	Limit for s ($\mu\text{g}/\text{L}$)	Range for x ($\mu\text{g}/\text{L}$)	Range p, p(s) (%)
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

(s) = Standard deviation of four recovery measurements, in $\mu\text{g}/\text{L}$

(x) = Average recovery for four recovery measurements, in $\mu\text{g}/\text{L}$

(p, p(s)) = Measured percent recovery

(D) = Detected; result must be greater than zero

(a) = Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop method performance data. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

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Attachment VII - BNA Poor Performing Compounds

The following compounds are considered to be poor performing compounds.

Pyridine
Aniline
Benzoic Acid
n-Nitrosodimethylamine
Hexachlorocyclopentadiene
4-Chloroaniline
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
2,4-Dinitro-2-methylphenol
Pentachlorophenol
Carbazole
Benzidine
Atrazine
Acetophenone
Caprolactam
Benzaldehyde
1,2,4,5-Tetrachlorobenzene

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SOP/Document REVISION FORM

02/06/07 R.1.0

SOP/DOC#	330345	Current revision date & number:	11/23/10 R13
Procedure/Method: Semi-Volatile organics by Gas Chromatography/Mass Spectrometry Using Capillary Column...			

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
12/10/10	cmj	2.1	Include SOP 330709 for 3511 extraction.		<i>cmj</i>	<i>cmj</i>
	cmj	2.31	Add definition for LVI			
	cmj	4.2	Include SOP 330709 for 3511 extraction			
	cmj	6.1.4	Add column 3 - Zebron 20mx.18mmx.36um film ZB5-MS Ultra Inert (or equivalent)			
	cmj	6.2	for LVI...up to 250ul could be injected.			
	cmj	7.8.3	for LVI...for PAH a 16ug/mL is made from the 800ug/mL Stock standard making the final concentration of 160ug/L. For SIM a 4ug/mL is made from the 800ug/mL Stock standard making a final concentration of 40ug/L.			
	cmj	7.9.2	for LVI...for PAH prep an Intermediate at 800ug/L from the stock mix described in section 7.6. For SIM prep a 2000ug/L serial intermediate then a 200ug/L intermediate.			

*Comments: Reason is addition of LVI Methodology to PAH and PAHSIM analysis of water samples.

Environmental Science Corporation
SOP/Document REVISION FORM

02/06/07 R.1.0

SOP/DOC#	330345	Current revision date & number:	11/23/10 R13
Procedure/Method: Semi-Volatile organics by Gas Chromatography/Mass Spectrometry Using Capillary Column...			

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
12/10/10	cmj	7.10.1	See attachment for curve levels for PAH and SIM by LVI.		<i>CM</i>	<i>DAW</i>
					<i>1</i>	<i>1</i>
	cmj	7.11	for LVI a standard is made at 2ppm from the stock 50ppm solution.			
	cmj	7.13	add SOP 330709 for 3511 extraction			
	cmj	8.2	For best results and detection limits the use of Autotune and Method 525.2 Limits (table 3) for tuning the instrument.			
	cmj	8.5.3	for LVI...PAH are injected with 200ppb of surrogate while SIM are injected at 40ppb.			
					<i>CM</i> 3/11/11	<i>DAW</i>
						<i>DAW</i>

*Comments: Reason is addition of LVI Methodology to PAH and PAHSIM analysis of water samples.

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FOR PAH by LVI calibration				
SVOC mix (800ppb) μ L	ISTD mix (uls of 16ppm)	Final Volume	Final conc. (ppb)	Level
5	10	1.0 mL	4	1
25	10	1.0 mL	20	2
50	10	1.0 mL	40	3
125	10	1.0 mL	100	4
200	10	1.0 mL	160	5
500	10	1.0 mL	400	6
750	10	1.0 mL	600	7
1000	10	1.0 mL	800	8

FOR PAHSIM by LVI calibration				
SVOC mix (200ppb) μ L	ISTD mix (uls of 4ppm)	Final Volume	Final conc. (ppb)	Level
5	10	1.0 mL	1	1
25	10	1.0 mL	5	2
50	10	1.0 mL	10	3
100	10	1.0 mL	20	4
200	10	1.0 mL	40	5
400	10	1.0 mL	80	6
1000	10	1.0 mL	200	7

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**TITLE: DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY ICP-AES
(EPA METHODS 6010B, 6010C, AND 200.7) INCLUDING HARDNESS (EPA METHODS 200.7
AND 6010B/C AND SM 2340B, 20TH EDITION) AND INDUSTRIAL HYGIENE SAMPLES (NIOSH
7300, 7301, AND 7303 AND OSHA ID-125G)**

SOP NUMBER: 340386

Prepared by: Jim Burns

Reviewed by: Jim Burns/Fran Meenan/Dixie Marlin

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Department Manager

QA Department

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP# 340386, Revision 14 (2/02/09).

- 1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals and some non-metals in solution. This procedure follows the guidelines established in EPA method 200.7 and SW-846 Method 6010B and 6010C for drinking water, waste water, ground water, TCLP, SPLP, and STLC leachates, soils, sludge, sediments, solid wastes, oils, and other extracts after appropriate digestion procedure is performed.

This procedure is also applicable to reporting calculated values for Calcium, Magnesium, and Total Hardness from values determined using EPA methods 200.7 or 6010B/C from groundwater, wastewater and drinking waters. Reporting limits for Hardness are derived from the annual MDL studies for Calcium and Magnesium of the appropriate determinative EPA method. The routine reporting limits for each category of hardness are listed below:

This procedure is also utilized in the analysis of industrial hygiene samples (i.e. air, paint, wipes and bulk materials) using NIOSH 7300, 7301, 7303 and/or OSHA ID-125G for various trace metals.

- 1.2 This method is applicable for the analytes listed in Table 1.2a, b and c. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix, and instrument operating conditions. Table 1.2 also lists the Reporting Limits (RLs), used routinely by ESC.

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**TITLE: DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY ICP-AES
(EPA METHODS 6010B, 6010C, AND 200.7) INCLUDING HARDNESS (EPA METHODS 200.7
AND 6010B/C AND SM 2340B, 20TH EDITION) AND INDUSTRIAL HYGIENE SAMPLES (NIOSH
7300, 7301, AND 7303 AND OSHA ID-125G)**

Table 1.2a: Environmental Analytes and Reporting Limits

Analyte	Aqueous				Sediment		
	WasteWater 6010B/C/200.7	Drinking Water 200.7	RL	Units	Solids 6010B/C	RL	Units
Aluminum	✓	✓	0.100	mg/L	✓	5.00	mg/Kg
Antimony	✓	✓	0.020	mg/L	✓	1.00	mg/Kg
Arsenic	✓	✓	0.020	mg/L	✓	1.00	mg/Kg
Barium	✓	✓	0.005	mg/L	✓	0.25	mg/Kg
Beryllium	✓	✓	0.002	mg/L	✓	0.05	mg/Kg
Boron	✓		0.200	mg/L	✓	10.0	mg/Kg
Cadmium	✓	✓	0.005	mg/L	✓	0.25	mg/Kg
Calcium	✓	✓	0.500	mg/L	✓	25.0	mg/Kg
Chromium	✓	✓	0.010	mg/L	✓	0.50	mg/Kg
Cobalt	✓	✓	0.010	mg/L	✓	0.50	mg/Kg
Copper	✓	✓	0.020	mg/L	✓	1.00	mg/Kg
Iron	✓	✓	0.100	mg/L	✓	5.00	mg/Kg
Lead	✓		0.005	mg/L	✓	0.25	mg/Kg
Lithium	✓	✓	0.015	mg/L	✓	0.75	mg/Kg
Magnesium	✓	✓	0.100	mg/L	✓	5.00	mg/Kg
Manganese	✓	✓	0.010	mg/L	✓	0.50	mg/Kg
Molybdenum	✓	✓	0.005	mg/L	✓	0.25	mg/Kg
Nickel	✓	✓	0.020	mg/L	✓	1.00	mg/Kg
Potassium	✓	✓	0.500	mg/L	✓	25.0	mg/Kg
Selenium	✓		0.020	mg/L	✓	1.00	mg/Kg
Silicon	✓		0.200	mg/L	✓	10.0	mg/Kg
Silver	✓	✓	0.010	mg/L	✓	0.50	mg/Kg
Sulfur	✓	✓	1.000	mg/L	✓	50.0	mg/Kg
Sodium	✓	✓	0.500	mg/L	✓	25.0	mg/Kg
Strontium	✓	✓	0.010	mg/L	✓	0.50	mg/Kg
Thallium	✓		0.020	mg/L	✓	1.00	mg/Kg
Tin	✓	✓	0.020	mg/L	✓	1.00	mg/Kg
Titanium	✓	✓	0.010	mg/L	✓	0.50	mg/Kg
Vanadium	✓	✓	0.010	mg/L	✓	0.50	mg/Kg
Zinc	✓	✓	0.030	mg/L	✓	1.50	mg/Kg

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7300, 7301, AND 7303 AND OSHA ID-125G)**

Table 1.2b: Industrial Hygiene Analytes and Reporting Limits

Analyte	Industrial Hygiene			
	Air Filters 6010B/C/NIOSH 7300/7301/7303	Welding Fumes (NIOSH 7300 or OSHA ID-125G)	RL	Units
Arsenic	✓		2.5	ug/sample
Barium	✓		2.5	ug/sample
Beryllium	✓		2.5	ug/sample
Cadmium	✓	✓	2.5	ug/sample
Chromium	✓	✓	2.5	ug/sample
Cobalt	✓		2.5	ug/sample
Copper	✓	✓	2.5	ug/sample
Iron	✓	✓	2.5	ug/sample
Lead	✓		2.5	ug/sample
Manganese	✓	✓	2.5	ug/sample
Molybdenum	✓		2.5	ug/sample
Nickel	✓	✓	2.5	ug/sample
Selenium	✓		2.5	ug/sample
Silver	✓	✓	2.5	ug/sample
Thallium	✓		2.5	ug/sample
Tin	✓		2.5	ug/sample
Titanium	✓		2.5	ug/sample
Vanadium	✓		2.5	ug/sample
Zinc	✓	✓	2.5	ug/sample

Table 1.2c: Hardness Categories and Reporting Limits

Hardness:	RL (mg/L)
Calcium Hardness	1.2
Magnesium Hardness	0.41
Total Hardness	1.6

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- 1.3 For the determination of total recoverable analytes in aqueous and solid samples an acid digestion process is required. Environmental samples for analysis by Method 6010B or 6010C including, TCLP or EP leachates, soils, sludge, sediments, and other solid wastes require an acid digestion prior to analysis. Samples are digested by SW-846 methods 3005 (Acid Digestion of Waters for Total Recoverable Metals), 3010 (Acid Digestion of Aqueous Samples), 3015 (Microwave Digestion of Aqueous Samples), 3050 (Acid Digestion of Sediments, Sludge, Soil, and Oils) and 3051 (Microwave Assisted Digestion of Sediments, Sludge, Soil, and Oils). Digestion methods are found in ESC SOPs 340388 and 340389.

The digestion of industrial hygiene samples is accomplished using ESC SOP# 340701, 340702, 340703.

- 1.4 The Clean Water Act has approved EPA Method 200.7 for demonstrating compliance on discharge monitoring for NPDES (National Pollution Discharge Elimination System) permits. 40 CFR136.3 has Guidelines for Establishing Test Procedures for Analysis of Pollutants. The National Primary Drinking Water Regulations for inorganic chemical sampling and analytical requirements can be found in 40 CFR141.23. Updates to these regulations can be found in the current Code of the Federal Register.
- 1.5. To determine dissolved analytes in aqueous samples, a 0.45µm filtration method is employed then the filtered samples are acidified. To reduce potential interferences, dissolved solids must be < 0.2% (w/v).
- 1.6 Analysis without acid digestion can be used for drinking water samples if the samples have been properly preserved with acid and have turbidity of < 1 NTU at the time of analysis. These samples must be acidified to match the acid matrix of the calibration standards and analyzed directly. This total recoverable determination procedure is referred to as "direct analysis". Silver concentration cannot be determined from direct analysis when chloride ions are present as a silver chloride precipitate may be formed. The sample must be acid digested to form a soluble silver chloride complex. Some primary drinking water metal contaminants may require sample concentration to meet regulatory drinking water reporting limits criteria.
- 1.8 When determining boron and silicon in aqueous samples, only plastic, PTFE (Teflon[®]) sample containers and laboratory glassware must be used. For accurate determination of boron in solid samples, only quartz or PTFE tubes must be used during acid digestion with immediate transfer of an aliquot of the final volume of digestate to a plastic centrifuge tube.
- 1.9 For the determination of titanium, white plastic and white printed containers must be avoided as titanium dioxide is used as a white pigment.

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- 1.10 The total recoverable sample digestion procedure dissolves and maintains in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis must be completed as soon as possible following sample preparation.
- 1.11 Detection limits and linear ranges for the elements vary with the wavelength selected, the spectrometer, and the matrix. Table 1.11 provides a list of routinely used wavelengths and the type of spectrometer view used

TABLE 1.11: WAVELENGTHS
(exact wavelengths vary slightly depending on the instrument)

Analyte	Wavelength (nm)	Type of View
Aluminum	308.215	Axial
Antimony	206.836	Axial
Arsenic	188.979	Axial
Barium	233.527	Axial
Beryllium	313.107	Axial
Boron	249.772	Axial
Cadmium	214.440	Axial
Calcium	315.887	Axial
Chromium	205.560	Axial
Cobalt	228.616	Axial
Copper	324.752	Axial
Iron	238.863	Axial
Lead	220.353	Axial
Lithium	670.784	Radial
Magnesium	279.077	Axial
Manganese	257.610	Axial
Molybdenum	202.031	Axial
Nickel	232.003	Axial
Potassium	766.490	Radial
Selenium	196.026	Axial
Silicon	251.611	Axial
Silver	328.068	Axial
Sodium	589.592	Radial
Strontium	407.771	Radial
Sulfur	181.972	Axial
Thallium	190.801	Axial

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Analyte	Wavelength (nm)	Type of View
Tin	189.927	Axial
Titanium	334.940	Axial
Vanadium	292.402	Axial
Zinc	213.857	Axial

- 1.12 Users of the data generated using this method must state the data-quality objectives (DQOs) prior to analysis.
- 1.13 Any deviations from this SOP must be documented. Deviations are reflected in a case narrative and the method is reported as modified. Per customer requirement, the procedure and QC criteria described in this SOP can be changed/modified. Authorization from the Technical Director and Project Manager is required for each modification and QA approval must also be secured for any deviation.
- 1.14 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are stored and maintained on file on the companywide intranet, under metals QC, then MDL folder.
- 1.15 Linear Dynamic Range (LDR) and Inter-element correction factor (IEC) studies must be analyzed once per year for each analytical instrument or when there are major changes/repairs to the instrument. Instrument Detection Limit studies must be analyzed at least quarterly for each analytical instrument.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The analysis described in this method involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are aspirated into the nebulizer and the resulting aerosol is transported to the plasma torch. The emission spectra are dispersed by a grating spectrometer separating the light emitted into the distinct wavelengths generated by each element in the sample. A photosensitive device monitors the intensities of each wavelength line in the spectra. The intensity of light on the photosensitive device produces a signal that is measured and processed by a computer system. Due to the many possible wavelengths of light generated by each element and possible overlapping of high intensity peaks, a background correction technique is required for trace element determination. Background intensities must be measured adjacent to the analyte spectra lines during analysis. The position selected for background intensity measurement can be selected on either or both sides of the analyte wavelength line and

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must be determined by the complexity of the spectrum adjacent to the analyte line. The position used for background correction must be as free from spectral interference as possible and must reflect the same change in background intensity as occurs at the analyte wavelength. Background correction is not required in cases of line broadening where the background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

- 2.3 Calibration Standard (CAL) – A series of known standard solutions used by the analyst for calibration of the instrument (i.e. preparation of analytic curve.)
 - 2.4 Dissolved Analyte - The concentration of analyte in an aqueous sample that has been passed through a 0.45µm membrane filter assembly prior to sample acidification and digestion.
 - 2.5 Total metals – The concentration of metals determined after sample digestion in field samples that have not been previously filtered.
 - 2.6 Instrument Detection Limit (IDL) - The instrument detection limit is calculated by performing ten sequential replicate measurements of a method blank. The standard deviation of these measurements is calculated and the IDL is equal to three times the standard deviation of the measurements. The IDL assures with 99% certainty that a value is above the instrument noise level.
- Note:** An IDL is a statistical determination without analytes present and an MDL is determined with low levels of analytes present.
- 2.7 Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration is linear.
 - 2.8 Plasma Solution - A solution that is used to determine the optimum torch height relative to the radio frequency (RF) coil for viewing the spectrum.
 - 2.9 Interference Check Sample (ICS) – A series of two solutions (ICSA & ICSAB) to verify that inter-element interferences are correctly compensated. The ICSA and ICSAB provide an adequate on-going test of inter-element correction (IEC) factors. These standards are referred to the Spectra Interference Check (SIC) in EPA Method 200.7
 - 2.9.1 ICSA – A solution containing only the interfering analytes at high concentrations.
 - 2.9.2 ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.

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- 2.10 **Water Sample** - For the purpose of this method, a sample taken from one of the following sources: drinking water, surface water, ground water, storm water, industrial or domestic wastewater.
- 2.11 **Preparation Batch** - For method 6010: A group of samples (not to exceed twenty) of a similar matrix, which have been digested at the same time using the same digestion process and have all necessary QC associated with them. For method 200.7: A group of samples (not to exceed ten) of similar matrix, which have been digested at the same time using the same digestion process and have all necessary associated QC.
- 2.12 **Analytical batch** - A group of samples that are analyzed in the same sequence with all appropriate preparation and analytical QC.
- 2.13 **Initial Calibration Verification (ICV)** – Analytical standard analyzed after the initial calibration to verify the initial calibration of the system. The ICV is prepared from a second source than that used to prepare the CAL standards.
 - 2.13.1 **Initial Calibration Verification - Low Level (ICVLL)** - Analytical standard analyzed after the initial calibration to verify the initial calibration of the system. The ICVLL is prepared from a second source than that used to prepare the CAL standards.
- 2.14 **Continuing Calibration Verification (CCV)** – Analytical standard run after every 10 samples to verify the continuing calibration stability of the system. The CCV is prepared from the same source as the standards used in the initial calibration. The CCV may not be used to replace the ICV.
 - 2.14.1 **Continuing Calibration Verification - Low Level (CCVLL)** – Analytical standard run after every 10 samples to verify the continuing calibration stability of the system. The CCVLL is prepared from a second source than that used to prepare the initial calibration.
- 2.15 **Initial Calibration Blank (ICB)** - a blank that is analyzed immediately after ICV. The ICB is a contamination and memory effect check solution.
- 2.16 **Continuing Calibration Blank (CCB)** - a blank that is analyzed immediately after every CCV. The CCB is a contamination and memory effect check solution.
- 2.17 **Inter-element correction (IEC) coefficient** - analyte concentration equivalent arising from a given interferent's concentration.

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- 2.18 **Reporting Limit (RL)** - Routinely the reporting limit is the lowest standard of the calibration curve. Technically, the reporting limit is the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions.
- 2.19 **Instrument Detection Limit (IDL)** - the concentration equivalent to the signal equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal.
- 2.20 **Serial Dilution** - a dilution and reanalysis of a field sample that is performed once per batch of samples. One sample is diluted 5X and reanalyzed.
- 2.21 **Post Spike** – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch.
- 2.22 **Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)** – Duplicate aliquots of a control sample of known composition. The LCS/LCSD are prepared and analyzed exactly like a field sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements in a clean matrix. Method precision can be determined using the results of the LCS/LCSD analysis. LCS/LCSD for air and wipe samples must be performed on a matrix that most closely matches that of the field samples being analyzed.
- 2.23 **Laboratory Fortified Blank (LFB), and Quality Control Sample (QCS)**, is used in method 200.7. This is the same as LCS (Laboratory Control Sample) stated above.
- 2.24 **Duplicate** – A second aliquot of a field sample analyzed using identical preparation and analytical procedures. Analysis of a sample duplicate monitors precision associated with laboratory procedures.
- 2.25 **Matrix Spike (MS) / Matrix Spike Duplicate (MSD)** - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine the appropriateness of the method for the field matrix by measuring recovery of target analytes. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.

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2.26 Internal Standard – An internal standard is an element which:

- Is not naturally present in samples
- By its addition does not contaminate the samples
- Is chemically compatible with the samples
- Does not introduce spectral interferences

An internal standard is used for three main reasons:

- Drift correction
- Accuracy improvement
- Repeatability improvement

If signal variation results from the sample introduction system (samples of different viscosity, matrix constitution), all the elements are corrected in the same way by use of a single internal standard. If variation results from a variation of the energy transfer, the internal standard most accurately corrects elements of similar energy.

2.27 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the MDL.

2.28 Surrogate - A compound that is similar to the target analytes in chemical composition and behavior and not expected to occur naturally in field samples. These analytes are spiked by preparation/analytical personnel to assess sample preparation and analytical efficiency in each individual field sample. This is also used as a check to ensure the autosampler actually hit the vessel being analyzed. If there is no recovery for the surrogate, chances are good that the autosampler probe missed the vessel.

2.29 Soil Sample – Non-aqueous materials ranging from municipal to industrial wastes that contain complex and sometimes hazardous substances.

2.30 Leachate – A liquid that has passed through or emerged from solid waste, collecting contaminants as it trickles through wastes, agricultural pesticides or fertilizers.

2.31 Method Detection Limit (MDL) - The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero.

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- 2.32 **Initial Demonstration of Capability (IDOC)** - A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank, laboratory control sample, etc. or the demonstration of capability must be repeated. See also Continuing Demonstration of Capability (CDOC).
- 2.32.1 The American Industrial Hygiene Association has specific requirements for demonstrations of competency for analysts performing work on client samples for environmental lead analysis (i.e. paint, wipes, filters, bulk materials). See additional guidance in ESC SOP# 030205, *Technical Training and Personnel Qualifications*.
- 2.33 **Continuing Demonstration of Capability (CDOC)** – At least annual verification of analyst continued ability to perform method acceptably.
- 2.34 **Linear Regression** - Mathematical technique for finding the straight line that best-fits the values of a linear function, plotted on a scatter graph as data points. If a 'best fit' line is found, it can be used as the basis for estimating the future values of the function by extending it while maintaining its slope.
- 2.35 **Reporting Limit Verification (RLV)** – A standard analyzed following initial calibration/calibration verification at or below the analyte concentration of the routine reporting level. It is analyzed per regulatory/method requirements for drinking water analyses and various other state/national regulatory programs to verify the accuracy of field sample results at the reporting level.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical should be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact could occur, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

Information on the known health hazards of the chemicals used in the laboratory can be found in the Material Safety Data Sheet (MSDS) book located in the hall outside the metals laboratory.

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- 3.2 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
 - 3.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.
 - 3.4 It is the responsibility of the user of this method to comply with relevant waste disposal practices and regulations.
 - 3.5 All ICP instruments provide protection from ultraviolet light emission. These shielding screens cannot be removed.
 - 3.6 Quarterly monitoring for lead contamination in areas performing lead sample preparation and analysis is performed as required for the American Industrial Hygiene Association's (AIHA) Environmental Lead Laboratory Accreditation Program (ELLAP). See ESC SOP# 340706, *Quarterly Monitoring for Lead Contamination*.
- 4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE
- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
 - 4.2 Prior to the collection of an aqueous sample, consideration must be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pre-treatment steps can be taken. The pH of all aqueous samples must be assessed immediately prior to sample digestion or "direct analysis" to ensure the sample has been properly preserved. If the field sample is properly preserved, the sample can be held up to 6 months prior to analysis.
 - 4.3 For the determination of dissolved elements, the sample must be filtered through a 0.45µm pore diameter membrane filter to remove the suspended elements or particles. This filtration must take place at the time of collection or as soon thereafter as practically possible. Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus must be used when the determinations of boron and silica are critical. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1:1) nitric acid: water immediately following filtration to pH < 2.

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**TITLE: DETERMINATION OF METALS AND TRACE ELEMENTS IN VARIOUS MATRICES BY ICP-AES
(EPA METHODS 6010B, 6010C, AND 200.7) INCLUDING HARDNESS (EPA METHODS 200.7
AND 6010B/C AND SM 2340B, 20TH EDITION) AND INDUSTRIAL HYGIENE SAMPLES (NIOSH
7300, 7301, AND 7303 AND OSHA ID-125G)**

- 4.4 For the determination of total recoverable elements in aqueous samples, samples must not be filtered, but acidified with (1:1) nitric acid: water to pH < 2. Preservation may be done at the time of collection, however, to avoid the hazards of strong acid use in the field, possible transport restrictions, or possible contamination, it is recommended that the samples be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample must be mixed and equilibrated for 24 hours. The pH is verified at < 2 prior to withdrawing an aliquot for acid digestion or "direct analysis". If, for reasons such as high alkalinity, the sample pH is verified to be >2, more acid must be added and the sample equilibrated for another sixteen hours until verified to be pH <2.
- 4.5 Solid samples require no chemical preservation prior to analysis. Store at 4° ± 2°C following sampling. Solid samples can be held up to six months from the time of sample collection until preparation and analysis.
- 4.6 For aqueous samples, a field blank must be prepared and analyzed as required by the data user. Use the same container and preservative as is used in field sample collection. The sample holding time is 6 months from the date and time of collection until analysis. Samples are preserved to pH <2 with nitric acid and stored at 4 ± 2°C.
- 4.7 Industrial Hygiene sample matrices (i.e. air filters, dust wipes, and paint) require no chemical or environmental preservation prior to analysis.

5.0 INTERFERENCES

- 5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
 - 5.1.1 Subtracting the background emission determined by measurement(s) adjacent to the analyte wavelength peak can usually compensate for background emission and stray light. The location(s) selected for the measurement of background intensity is determined by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. Changes in background correction must be saved in the instrument method. Background correction can be established by scanning the following three solutions: 1) blank (same as calibration blank); 2) solution, containing analytes at significant concentration to raise a signal above background signal (CCV solution may be used) at mid-range of the curve; 3) solution(s) containing most common interfering elements at high concentration and other interferences as well (ICSAB solution may be used).

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- 5.1.2 Spectral overlaps can be compensated for by equations that correct for inter-element contributions, which involves measuring the interfering elements. When operative and uncorrected, these interferences produce false-positive determinations and be reported as analyte concentrations. Users may apply inter-element correction factors determined on their instruments within tested concentration ranges to compensate (offline or online) for the effects of interfering elements. Consult the method for specific identified interferences.
- 5.1.3 When inter-element corrections are applied, there is a need to verify their accuracy by analyzing spectral interference check solutions. The IEC's are established by analyzing a solution of the interfering element at concentration of 90% of the upper LDR limit, measuring the analyte concentration equivalents arising from the interfering element, calculating the interference factor as analyte reading in mg/L, then dividing by the interfering element concentration. The IEC's are changed in the stored ICP instrument method. Inter-element corrections vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples might contain uncommon elements that could contribute spectral interferences.
- 5.1.4 Interference effects must be evaluated for each individual instrument. For each instrument, intensities vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). To determine the appropriate location for offline background correction, the user must scan the area on either side of the peak adjacent to the wavelength and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free from offline inter-element spectral interference or a computer routine must be used for their automatic correction on all determinations.

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- 5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as using a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This can be controlled using a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision. This is accomplished with the use of mass flow controllers.
- 5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching, and by standards addition procedures. Chemical interferences are highly dependent on matrix type.
- 5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them.
- 5.5 Linear Dynamic Range (LDR) study is performed by analyzing a solution of each element at maximal concentration unless the result falls outside 10% RPD. The highest calibration standard cannot be greater than the LDR. If an interferent is found greater than the LDR and an IEC factor is established between the interferent and analyte of interest, the sample must be diluted for proper correction of inter-element interferences. Instrument methods with different calibration standard concentrations require separate LDR studies.
- 5.6 Background correction is performed as needed and LDR and IEC studies are completed at least annually or whenever significant changes to instrumentation are made. Background subtraction is not performed for environmental samples, but may be utilized for industrial hygiene (IH) analyses where the sample collection matrix imparts significant bias to the field sample analysis. If background subtraction is utilized for IH samples, a notation is included on the final report indicating that background subtraction has been performed.

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7300, 7301, AND 7303 AND OSHA ID-125G)**

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Inductively coupled plasma emission spectrometer:
 - 6.1.1 Perkin Elmer 4300 DV ICP, Perkin Elmer 5300 DV ICP, or equivalent, with background correction and computer control.
 - 6.1.2 Perkin Elmer or Cetac Autosampler
 - 6.1.3 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.
- 6.2 Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125mL to 1L capacities.
- 6.3 One-piece stem FEP wash bottle with screw closure, 125mL capacity.
- 6.4 Adjustable pipettes (Eppendorf or equivalent), ranges from 2µL to 5000µL.
- 6.5 Class A volumetric flasks for standards preparations.
- 6.6 Polypropylene (PP) conical tubes.
- 6.7 Peristaltic pump.

7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner if a problem is detected unless otherwise noted.
- 7.2 Hydrochloric acid, concentrated (sp. gr. 1.19) - HCl.
 - 7.2.1 Hydrochloric acid (1+1) - Add 500mL concentrated HCl to 400mL reagent water and dilute to 1L with reagent water.

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7.3 Nitric acid, concentrated (sp. gr. 1.41) - HNO₃.

7.3.1 Nitric acid (1+1) - Add 500mL concentrated HNO₃ to 400mL reagent water and dilute to 1L with reagent water.

7.4 Reagent water. All references to water in this method refer to ASTM Type I grade water.

7.5 Mixed Calibration Standard Solutions – All standards are prepared in Class A volumetric flasks using adjustable pipettes. The final acid concentration is 5% Nitric Acid. The following formula is used to calculate the amount of stock solution V_{stock} (mL) to use: V_{stock} = V_{std} * C_{std} / C_{stock}, where V_{std} (mL) is final volume of the standard to be prepared, C_{std} (mg/L) is concentration of the standard to be prepared, C_{stock} (mg/L), is concentration of the stock solution. The concentration of the lowest standard must be at or below the reporting limit. All information about prepared standards is stored in TREE, a standards/reagents tracking application stored on the internal ESC servers.

Environmental Express Custom Mixes, or equivalent, is used to make the following calibration solutions:

Note: Environmental Express Custom Mix #HP2988A contains the following elements and concentrations in $\mu\text{g/mL}$: Al-1000, As-100, Ba-100, Be-100, B-100, Cd-100, Ca-1000, Cr-100, Co-100, Cu-100, Fe-1000, Pb-100, Li-100, Mg-1000, Mn-100, Ni-100, P-100, K-1000, Se-100, Na-1000, Sr-100, Ti-100, V-100, Zn-100.

Environmental Express Custom Mix #HP2988B contains the following elements and concentrations in $\mu\text{g/mL}$: Sb-100, Mo-100, Si-100, Ag-10, Sn-100, Ti-100.

Environmental Express Custom Mix #HP100054-5 contains Sulfur at a concentration of 1000 $\mu\text{g/mL}$.

Calibration Standard	Amount of Stock used per 100mL of 5% nitric acid
Std 6	10mL Stock Custom Mix-HP2988 A&B
Std 5	1mL Stock Custom Mix-HP2988 A&B
Std 4	1mL of 10ppm std (Std 6)
Std 3	1mL of 1ppm std (Std 5)
Std 2	0.5mL of 1ppm std (Std 5)
Std 1	2mL of 0.1ppm std (Std 4)

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Concentration of Target Analytes in Calibration Standards in mg/L.

Analyte	Std1	Std2	Std3	Std4	Std 7	Std5	Std 6
Silver				0.010		0.100	1.00
Aluminum			0.100	1.000		10.0	100.
Arsenic			0.010	0.100		1.000	10.0
Boron			0.010	0.100		1.000	10.0
Barium		0.005	0.010	0.100		1.000	10.0
Beryllium	0.002	0.005	0.010	0.100		1.000	10.0
Calcium			0.100	1.000		10.0	100.
Cadmium		0.005	0.010	0.100		1.000	10.0
Cobalt			0.010	0.100		1.000	10.0
Chromium			0.010	0.100		1.000	10.0
Copper			0.010	0.100		1.000	10.0
Iron			0.100	1.000		10.0	100.
Potassium				1.000	0.500	10.0	100.
Lithium		0.005	0.100	0.100		1.000	10.0
Magnesium			0.100	1.000		10.0	100.
Manganese			0.010	0.100		1.000	10.0
Molybdenum	0.002	0.005	0.010	0.100		1.000	10.0
Sodium				1.000	0.500	10.0	100.
Nickel			0.010	0.100		1.000	10.0
Lead		0.005	0.010	0.100		1.000	10.0
Antimony			0.010	0.100		1.000	10.0
Selenium			0.010	0.100		1.000	10.0
Silicon			0.010	0.100		1.000	10.0
Strontium	0.002	0.005	0.010	0.100		1.000	10.0
Sulfur				1.000		10.0	100.
Tin			0.010	0.100		1.000	10.0
Thallium			0.010	0.100		1.000	10.0
Vanadium			0.010	0.100		1.000	10.0
Zinc			0.010	0.100		1.000	10.0
Titanium			0.010	0.100		1.000	10.0

NOTE: If the addition of silver to the recommended mixed-acid calibration standard results in an initial precipitation, add 15 mL of reagent water and warm the flask until the solution clears. For this acid combination, the silver concentration should be limited to 0.5mg/L.

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7.6 Blanks - Three types of blanks are required for ICP-AES analysis.

- 7.6.1** The calibration blank is used in establishing the analytical curve. The calibration blank for aqueous samples and extracts is prepared by acidifying reagent water to the same acid concentration as used for the standards. The calibration blank must be stored in a FEP bottle. Following calibration, the Initial Calibration Blank (ICB) is analyzed prior to sample analysis. A Continuing Calibration Blank (CCB) is analyzed following the CCV after every ten samples and at the end of the analytical sequence to verify on-going acceptable instrument conditions.
 - 7.6.2** The method blank is used to assess possible contamination from the sample preparation procedure. The method blank must contain all the reagents in the same volumes as used in sample preparation. The method blank must be prepared in the same manner as the samples including sample digestion, when applicable. Method blanks for air filters and dust wipes must contain a sampling matrix that most closely resembles the matrix used for the collection of field samples.
 - 7.6.3** The rinse blank is prepared by acidifying reagent water to the same concentrations as the acids as used in the calibration blank. This solution is stored in a convenient manner. The rinse blank is used for equipment "wash out" to flush the sample delivery system and eliminate memory effects (carryover) from previous samples or standards.
- 7.7** Initial Calibration Verification (ICV) – The ICV is an analytical standard solution from a second source different from the calibration and CCV standards. The ICV is prepared at a mid-range concentration within the linear working range of the instrument. The ICV must have the same acid matrix as the Calibration Standards, CCV, blanks and the samples.

ICV solutions are purchased through High Purity Standard SP2762-3227HPZ-A, High Purity Standard SP2762-3227HPZ-B, and Ultra Scientific ICP-016, or equivalents.

Custom Mix #SP2762-3227HPZ-A (stock) contains the following element and concentrations in **ug/mL**: Al-500, As-50, Ba-50, Be-50, B-50, Cd-50, Ca-500, Cr-50, Co-50, Cu-50, Fe-500, Pb-50, Li-50, Mg-500, Mn-50, Ni-50, P-50, K-500, Se-50, Na-500, Sr-20, Ti-50, V-50, Zn-50.

Custom Mix # SP2762-3227HPZ-B (stock) contains the following elements and concentrations in **ug/mL**: Sb-50, Mo-50, Si-50, Ag-5, Sn-50, Ti-50.

Sulfur, Ultra Scientific # ICP-016 at a concentration of 1000 **ug/mL**.

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This solution is prepared, by spiking 10 mL of the custom stock solution A and 10mL of the custom stock solution B into a 100mL volumetric flask, then adding 5mL of concentrated Nitric Acid and diluting to 100mL using DI water. The final concentration for this solution is 5ppm for the following elements: Sb, As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Li, Mn, Mo, Ni, P, Se, Si, Ti, Sn, Ti, V, and Zn. The final concentration for Al, Ca, Fe, Mg, K, and Na is at 50ppm. The final concentration for Ag is 0.5ppm and Sr is 2ppm. If sulfur is required, the concentration of the standard is 10ppm.

- 7.8 Continuing Calibration Verification (CCV) – The CCV is the mid-range calibration standard. The CCV is used to represent the ongoing calibration efficiency of the instrument and must be repeated following every ten samples and at the conclusion of the sequence. EPA Method 200.7 refers to this standard as the Instrument Performance Check (IPC) standard.

The CCV solution is prepared using the same instructions for the mid range standard from the solution used to make the calibration standards (Environmental Express Custom Mix #HP2988A&B). All analytes are present in the CCV solution at 5.0mg/L except aluminum, calcium, iron, magnesium, sodium, potassium, which are present at 50.0mg/L, and silver at 0.5mg/L. *The sulfur stock solution is a purchased from Environmental Express (Cat#: HP100054-5), or equivalent.

Analyte	Concentration (mg/L)		Analyte	Concentration (mg/L)
Silver	0.20		Manganese	0.20
Aluminum	2.00		Molybdenum	0.10
Arsenic	0.40		Sodium	10.0
Boron	4.00		Nickel	0.40
Barium	0.10		Lead	0.10
Beryllium	0.04		Antimony	0.40
Calcium	10.0		Selenium	0.40
Cadmium	0.10		Silicon	4.00
Cobalt	0.20		Strontium	0.20
Chromium	0.20		Tin	0.40
Copper	0.40		Thallium	0.40
Iron	2.00		Vanadium	0.20
Potassium	10.0		Zinc	0.60
Lithium	0.30		Titanium	0.20
Magnesium	2.00		Sulfur*	10.0

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- 7.9 Initial/Continuing Calibration Verification – Low Level (ICVLL/CCVLL) – The ICVLL/CCVLL is an analytical solution from a second standard source different from the calibration and CCV standards. The ICVLL/CCVLL is prepared at a low concentration within the linear working range of the instrument. The ICVLL/CCVLL must have the same acid matrix as the Calibration Standards, CCV, blanks and the samples.

The ICVLL/CCVLL stock solution is a custom made solution from Environmental Express (Cat#: HP3712-12), or equivalent, and contains the following analytes at the concentrations listed below. *The sulfur stock solution for this is a separate solution purchased from Ultra Scientific # ICP-016, or equivalent.

This working standard for the ICVLL/CCVLL is prepared by diluting 50mL of the solution(s) above into 1000mL of Di water that has been acidified to 5% Nitric Acid solution to matrix match the solution with the calibration standards and samples. The concentration of the working ICVLL/CCVLL solution is listed in the table below:

Analyte	Concentration (mg/L)		Analyte	Concentration (mg/L)
Silver	0.01		Manganese	0.01
Aluminum	0.1		Molybdenum	0.005
Arsenic	0.02		Sodium	0.50
Boron	0.2		Nickel	0.02
Barium	0.005		Lead	0.005
Beryllium	0.002		Antimony	0.02
Calcium	0.50		Selenium	0.02
Cadmium	0.005		Silicon	0.20
Cobalt	0.01		Strontium	0.01
Chromium	0.01		Tin	0.02
Copper	0.02		Thallium	0.02
Iron	0.1		Vanadium	0.01
Potassium	0.5		Zinc	0.03
Lithium	0.015		Titanium	0.01
Magnesium	0.10		Sulfur*	1.00

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- 7.10 Interference Check Solutions (ICSA and ICSAB) – The ICSA and ICSAB are prepared to contain known concentrations of interfering elements that provides a test of the correction factors. The ICSA solution contains the interfering elements at a high concentration and the ICSAB contains both the interfering analytes at a high concentration and the analytes of interest at 0.5 to 1.0mg/L. EPA Method 200.7 refers to this standard as the Spectral Interference Check (SIC) standard.
- 7.10.1 The ICSA solution contains 500mg/L of each Al, Ca, Mg, Na, 200 mg/L Fe and 10 mg/L K. This solution is prepared from a 1:10 dilution of purchased stock from Environmental Express, Catalog No. HP2740-1L.
- 7.10.2 The ICSAB solution contains all the components of the ICSA and the analytes of interest spiked at 0.50µg/mL or 1.00µg/mL. The solution is prepared from a 1:10 dilution of the stock from Section 7.10.1 (Environmental Express, Catalog No. HP2740-1L) and a 1:100 dilution of the purchased stock from Environmental Express, Catalog No. HP2739-1L.
- In the working solution, silver, boron, cadmium, nickel, lead, silica, and zinc are present at 1.0mg/L. Arsenic, barium, beryllium, cobalt, chromium, copper, manganese, molybdenum, antimony, selenium, thallium, titanium, tin, and vanadium are present at 0.5mg/L.
- 7.11 The aqueous laboratory control standard (LCSW) is purchased with all analytes at a concentration of 100µg/mL except calcium, magnesium, potassium, and sodium, which are at 1000µg/mL. The LCSW is purchased from Ultra Scientific, Number ICUS-726 or equivalent. This working solution is prepared by spiking 0.5mL of the Ultra Scientific Stock Solution then diluting to a 50mL final volume using DI water. The final concentration is 1ppm, except calcium, magnesium, potassium, and sodium, which are present at 10ppm.
- 7.12 The solid laboratory controls standard (LCSS) is purchased through Environmental Resources Associates, catalog No. PPS-46, or equivalent. Analytes are present at various concentrations. This is a purchased certified solid at varying analyte concentrations that change from one lot to the next.
- 7.13 The aqueous and solid matrix spike and matrix spike duplicate are prepared from the purchased standard specified in Section 7.10. The working solutions are prepared as directed in SOP 340389, Table 7.6.

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7300, 7301, AND 7303 AND OSHA ID-125G)**

- 7.14 Internal Standard (10,000µg/mL) – Yttrium is used as an internal standard and is added to all samples, standards, and extracts. The yttrium is prepared from a 10.00µg/mL purchased standard (Environmental Express or equivalent) and diluted to a final concentration of 5ppm as directed in SOP 340389, Table 7.6. The internal standard response is used to measure the relative responses of other method analytes in each sample. See the acceptance criteria in Section 10.13 of this procedure.
- 7.15 Plasma Solution - The plasma solution is used for determining the optimum viewing height of the plasma above the RF coil prior to using the method. The plasma stock solution is purchased from Environmental Express (or equivalent) at 1000µg/mL manganese. The solution is diluted to 1ppm manganese as directed in SOP 340389, Table 7.6 and stored in a FEP bottle. The solution is used according to the instrument manufacturer's instructions.
- 7.16 Surrogate – A standard scandium solution at 1000ppm is purchased from Environmental Express (or equivalent) and is diluted to a final concentration of 1.0 ppm as directed in SOP 349389, Table 7.6.
- 7.17 Lower limit of quantitation check sample: The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within historical laboratory accuracy limits of their true value. This check should be used to both establish and confirm the lowest quantitation limit.

The lower limits of quantitation determination using reagent water represents a best case situation and does not represent possible matrix effects of real-world samples. For the application of lower limits of quantitation on a project-specific basis with established data quality objectives, low-level matrix specific spike studies may provide data users with a more reliable indication of the actual method sensitivity and minimum detection capabilities.

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7300, 7301, AND 7303 AND OSHA ID-125G)**

8.0 PROCEDURE

8.1 Sample Analysis

- 8.1.1 Prior to daily calibration of the instrument, inspect the sample introduction system including the nebulizer, torch, injector tube for salt deposits, dirt, and debris that would restrict solution flow and affect instrument performance. Replace the uptake tubing daily. If any of the sample introduction parts appear soiled, first remove the part from the instrument by following the maintenance procedure in the instrument manual. Once removed, attempt to clean the part with a dilute solution of 5% nitric acid. Cleaning may be performed using a cotton swab or by submersing the part in the acid solution for no longer than 5 minutes. If cleaning is successful, dry the part using compressed air and replace it in the instrument. If cleaning does not adequately remove the residue, the part must be replaced with a new one in accordance with the manufacturer's directions. Replacement parts are kept in the cabinet in the instrument lab.
- 8.1.2 The instrument must be allowed to become thermally stable before calibration and analyses. This usually requires at least 60 minutes of operation. It is necessary to align the spectrometer with the plasma prior to analysis. This is accomplished by placing the sample probe in a plasma solution (Section 7.14). The plasma solution is used per instrument manufacturer's instructions. The Mn solution is aspirated for 60 seconds to allow enough time for the Mn to be present in the plasma. Once the Mn has been adequately aspirated, the spectrometer takes a series of Mn readings at various X and Y coordinates to find the optimum plasma viewing position. This is chosen by the combined X and Y coordinates that yield the highest detector response for Mn during the optimization procedure. Mn is used in optimization procedures because its wavelength (257.6nm) is mid-range in the spectrum.
- 8.1.3 For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank. The concentration of the lowest standard must be at or below the reporting limit. A peristaltic pump must be used to introduce all solutions, samples, and internal standard to the nebulizer. To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before accumulating data. Use the average value from three replicate integration periods of the signal to be correlated to the analyte concentration. Flush the system with the rinse blank for a minimum of 60 seconds between each standard. The calibration line is generated by first order linear regression from a calibration blank and six calibration standards where each element is present in at least three concentration levels. The blank is included as a point in the calibration curve, but the regression line is not "forced through zero". Calibration acceptance criteria are described in section 10.4.

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- 8.1.4 All standards/samples/QC etc. contain yttrium as the internal standard. The instrument adds yttrium automatically. A 5 ppm solution is prepared to determine an intensity baseline. The instrument injects a constant volume into each sample/standard and monitors the intensity at the sample level. Yttrium is the chosen alternative to the method of standard additions (MSA). If signal variation results from the sample introduction system (samples of different viscosity, matrix constitution), all the elements are corrected in the same way by using yttrium as a single internal standard. If variation results from a variation of the energy transfer, the internal standard most accurately corrects elements of similar energy. Internal standard acceptance criteria are described in section 10.13.

For the surrogate, add 0.1mL of 1000ppm scandium solution (Section 7.15) to the 1ppm calibration standard and adjust to volume. The acceptance criteria are described in section 10.14. The surrogate is used to ensure that the autosampler samples correctly from the vessel. If there is no surrogate recovery following analysis, chances are that the autosampler missed the vessel.

The instrument supplies triplicate readings for each solution analyzed. The average of the readings is used for quantitation.

- 8.1.5 After calibrating and rinsing the instrument, analyze the ICV/ICVLL standards. These standards are prepared as directed in section 7.7 and 7.9. Acceptance criteria are described in section 10.5.
- 8.1.6 Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV, CCVLL, and CCB that must meet the criteria described in section 10.6.
- 8.1.7 The interference check standards (ICSA and ICSAB) are used to verify the inter-element and background correction factors at the beginning and end of an analytical sequence or twice during every 8-hour work shift, whichever is more frequent. The interference check standards must meet the criteria found in section 10.10.
- 8.1.8 An Initial Calibration Blank (Section 7.6.1) is analyzed before sample analysis is initiated. Acceptance criteria are described in section 10.15.
- 8.1.9 After completion of the above calibration requirements, samples must be analyzed in the same operational manner used in the calibration routine with the rinse blank also being used between all sample solutions, method blanks, Laboratory Control Standards, matrix spike, matrix spike duplicates, and check solutions.

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8.1.10 If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte Linear Dynamic Range (LDR), see section 10.16 for further guidance.

9.0 DATA ANALYSIS AND CALCULATIONS

- 9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/Kg dry weight for solid samples. Industrial hygiene sample data is routinely reported in ug/sample and may be reported in additional units as required by the client. The mean of three "passes" from the spectrometer is used to derive sample concentrations.
- 9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with compensation for sample dilution. Do not report analyte concentrations below the MDL.
- 9.3 For total recoverable aqueous analytes, multiply solution analyte concentrations by the dilution factor 0.5 when a 100mL aliquot is used to produce the 50mL final digestate volume, and report data. If a different aliquot volume other than 100mL is used for sample preparation, adjust the dilution factor accordingly. Account for any additional dilution of the prepared sample digestate required to complete the determination of any analytes exceeding 90% or greater of the LDR upper limit. Do not report data below the determined analyte MDL concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required by the analysis.
- 9.4 For analytes with RLs < 0.01mg/L, round the data values to the thousandth place and report analyte concentrations up to three significant figures. For analytes with RLs >/- 0.01mg/L round the data values to the hundredth place and report analyte concentrations up to three significant figures. Analyte concentrations for solids data should be rounded in a similar manner before dry weight corrections in Section 9.5 are performed.
- 9.5 For total recoverable analytes in solid samples, round the solution analyte concentrations (mg/L) as instructed in Section 9.4. Report the data up to three significant figures as mg/Kg dry-weight basis unless specified otherwise by the program or data user. Calculate the concentration using the equation below and do not report analyte data below the estimated solids RL or an adjusted RL because of additional dilutions required to complete the analysis:

$$\text{Sample Conc. (mg/Kg)} = \frac{C \times V \times D}{W}$$

where: C = Concentration in extract (mg/L)
V = Volume of extract (L, 100 mL = 0.1L)
D = Dilution factor (undiluted = 1)
W = Weight in Kg of sample aliquot extracted (g x 0.001 = Kg)

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- 9.6 Soil samples are reported on a dry weight basis. Soil samples must be processed using the ESC SOP #340326, *Percent Moisture*. After a dry weight for each sample has been obtained, the calculations are performed as follows:

$$\% \text{ solids (S)} = \frac{\text{DW}}{\text{WW}} \times 100$$

where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

At ESC, dry weight reporting conversions are performed by the LIMS data system.

- 9.7 LCS/ICV/CCV Percent Recovery (%R):

$$\% R = \frac{\text{Measured concentration}}{\text{Actual concentration}} \times 100$$

- 9.8 Matrix Spike Recoveries (%R_{MS/MSD}):

$$\% R_{\text{MS/MSD}} = \frac{O_i - O_s}{T_i} \times 100$$

where: O_i = observed sample concentration with the spike added
O_s = the observed value for the sample without the spike
T_i = True value of the spike added

- 9.9 Relative Percent Difference (%RPD):

$$\text{RPD} = \frac{\text{Value 1} - \text{Value 2}}{\left(\frac{\text{Value 1} + \text{Value 2}}{2} \right)} \times 100$$

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9.10 Linear calibration model:

$$y = mx + b$$

where: y = Response A_x for External Standard
 x = Concentration C_x for External Standard
 m = Slope
 b = Intercept

Slope (m):

$$m = \frac{[(Swx_i * y_i) - (Swx_i * Swy_i)]}{[(Sw * Swx_i^2) - (Swx_i * Swx_i)]}$$

Intercept (b):

$$b = y_{AVE} - (m * (x_{AVE}))$$

Correlation Coefficient (r):

$$r = \frac{[(Sw * Swx_i * y_i) - (Swx_i * Swy_i)]}{\sqrt{[(Sw * Swx_i^2) - (Swx_i * Swx_i)] * [(Sw * Swy_i^2) - (Swy_i * Swy_i)]}}$$

Coefficient of Determination (r^2):

$$r^2 = r * r$$

Where: n = number of x, y pairs
 x_i = individual values for the independent variable
 y_i = individual values for the dependent variable
 w = weighting factor, for equal or no weighting $w = 1$
 x_{AVE} = average of the x values
 y_{AVE} = average of the y values
 S = the sum of all the individual values

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9.11 Hardness calculations:

$$\text{Total Hardness, mg equivalent CaCO}_3/\text{L} = 2.497 [\text{Ca, mg/L}] + 4.118 [\text{Mg, mg/L}]$$

$$\text{Calcium Hardness} = 2.497 [\text{Ca, mg/L}]$$

$$\text{Magnesium Hardness} = 4.118 [\text{Mg, mg/L}]$$

9.12 To calculate the silica concentration from silicon analysis:

$$\text{Silica (mg/L)} = 2.14 * [\text{Silicon, mg/L}]$$

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications*, before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated run log to record batch order and standards/reagents used during analysis. See SOP 030201, *Data Handling and Reporting*.

10.3 Batch Analyses:

10.3.1 Environmental Preparation Batches: Preparation batches are defined as sets of 1 - 20 samples, for Method 6010B, and 1-10 samples for Method 200.7. Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD) (the LCSD is only included if sufficient volume is not available for a Sample Duplicate), 1 Sample Duplicate (DUP), if field sample volume permits, 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information must be maintained in the preparation documentation assigned to the department.

Industrial Preparation Batches (Air filters and dust wipes): Preparation batches are defined as sets of 1 - 20 samples, for NIOSH Methods 7300, 7301, 7303 and OSHA Method ID-125G. Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD). All batch information must be maintained in the preparation documentation assigned to the department.

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Industrial Hygiene Preparation Batches (Lead paint and bulk materials):
Preparation batches are defined as sets of 1 - 20 samples, for EPA 6010B/C.
Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Sample Duplicate (DUP), if field sample volume permits, and 1 Matrix Spike/Spike Duplicate (MS/MSD) pair, if sufficient field sample volume permits. All batch information must be maintained in the preparation documentation assigned to the department

- 10.3.2 Analytical Batches: Analytical batches are defined as a sequence of samples analyzed concurrently using the same calibrated instrument. Analytical batches include the QC samples produced in the Preparation Batches, in addition to: 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Initial Calibration Verification (ICV) following initial calibration, 1 Initial Calibration Verification-Low Level (ICVLL) following initial calibration 1 Initial Calibration Blank following the ICVLL, 1 Continuing Calibration Verification (CCV) following each 10 samples and at the conclusion of the sequence, 1 Continuing Calibration Verification-Low Level (CCVLL) following each 10 samples and at the conclusion of the sequence, 1 Continuing Calibration Blank (CCB) following each CCV, 1 Interference Check Sample A (ICSA) and 1 Interference Check Sample AB (ICSAB) following each initial calibration and at the end of the sequence or at least twice per each 8 hour shift. All batch information must be maintained in the analytical documentation assigned to the department.
- 10.4 Initial Calibration - Run a calibration curve on a daily basis that employs a minimum of a calibration blank and six standards where each element is present in at least three of the standard levels. For EPA Methods 200.7 & 6010B, the linear regression correlation coefficient for the each analyte in the calibration curve lines must be 0.995 or better. For EPA Method 6010C, the regression correlation coefficient must be 0.998 or better. If the correlation coefficient does not meet the acceptance criteria, see the corrective action guidance listed in section 11.1.
- 10.5 Initial Calibration Verification (ICV/ICVLL) - Verify the initial instrument standardization by analyzing appropriate check standards following calibration.
- 10.5.1 **EPA Methods 6010B/C & 200.7** - The mid-level ICV standard recovery results must be $\pm 10\%$ of the true value for EPA methods 6010B, and $\pm 5\%$ of the true value for EPA method 200.7. The RSD must be within 3% for the triplicate passes of the spectrometer. If the RSD exceeds 3%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

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- 10.5.2 **EPA Method 6010C only** - The mid-level ICV standard recovery results must be ±10% of the true value for EPA methods 6010C, the RSD must be within 3% for the triplicate passes of the spectrometer. The low-level ICV standard recovery results should be within historical laboratory accuracy limits. The RSD must be within 3% for the triplicate passes of the spectrometer. If the RSD exceeds 3%, locate and correct the cause of the problem and do not proceed until this criterion is met.
- 10.5.3 Corrective actions for failures can be found in section 11.2.
- 10.6 Continuing Calibration Verification (CCV/CCVLL) - Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV that must meet the following criteria:
- 10.6.1 **For SW-846 Method 6010B** – Continuing Calibration Verification (CCV) recovery must be within 90%-110%. Instrument calibration verification is achieved by analyzing both a CCV standard and a CCB (instrument blank). CCV recovery must be 90-110% of the known concentration.
- 10.6.2 **For EPA Method 200.7** - Continuing calibration verification (CCV) analyzed after the calibration and before sample analysis must have a recovery of 90-110%. For all subsequent continuing calibration standards (CCV's), the recovery limits are 90%-110%.
- 10.6.3 **For SW-846 Method 6010C** - Instrument calibration verification for method 6010C is achieved by analyzing both a mid-level CCV standard, a low-level CCV standard, and a CCB (instrument blank). The mid-level Continuing Calibration Verification (CCV) recovery must be within 90%-110%. The low-level Continuing Calibration Verification (LLCCV) recovery should be within historical laboratory accuracy limits.
- 10.6.4 If these criteria are not met, corrective actions are found in section 11.3.
- 10.7 Method Blank - A method blank is generated for each analytical batch. The method blank accompanies each sample batch throughout the sample preparation and analysis procedure to determine if any contamination is introduced during sample processing. A method blank is a volume of reagent water that is carried through the entire digestion and analysis procedure with the samples. The method blank must not contain analytes >½ RL or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. If target analytes are present in the method blank, corrective action must be taken. See section 11.5 for corrective actions.

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- 10.8 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** - A matrix/matrix spike duplicate must be prepared for each matrix for each batch of 10 samples for method 200.7 or 20 samples for method 6010B/C, where sufficient sample volume was submitted by the client. Matrix spike and matrix spike duplicate are prepared from a sample aliquot spiked with the known concentration of analytes.

The matrix spike recoveries must meet the criteria in the table below unless the analyte concentration in the sample is at least 4 times greater than the spike concentration.

Method	Acceptance Criteria	
	Water	Soil
6010B & 6010C	75 – 125%	75 – 125%
200.7	75 – 125%	NA

Assess that the matrix spike duplicate precision (%RPD) results meet project-established goals acceptance criteria. If no project goals are specified, then results for the RPD must be less than 20%.

See section 11.9 for corrective actions associated with MS/MSD.

- 10.9 **Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)** - A LCS must be digested and analyzed with each batch of 10 samples for method 200.7 or 20 samples for method 6010B. The LCS recovery must be 85-115%. The LCSD is only performed if insufficient sample was submitted by the client for a duplicate, matrix spike and matrix spike duplicate pair. In those cases, the accuracy criteria for the LCSD is the same as for the LCS and the RPD must be less than 20%.

See section 11.12 for corrective actions associated with LCS/LCSD.

- 10.10 **Interference Check Standards (ICSA/ICSAB)** - The recovery of the ICSAB elements must be 80-120%. The recovery of the ICSAB elements must be 80-120%. If the results are unsatisfactory, see section 11.4 for further guidance. Do not proceed until this criterion is met.

- 10.11 **Serial Dilution** - If the analyte concentration is sufficiently high, an analysis of a 1:4 dilution must agree within 10% of the original determination. If not, see section 11.11 for further guidance.

- 10.12 **Post digestion Spike** - An analyte spike added to a portion of a prepared sample, or its dilution and must be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, see section 11.11 for further guidance.

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7300, 7301, AND 7303 AND OSHA ID-125G)**

- 10.13 Internal Standard - Verify the internal standard responses. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample must fall within 70-125% of the response in the calibration blank. If the %RI of the response in the sample falls outside of these limits, see section 11.8 for further guidance.
- 10.14 Surrogate - Verify the presence of the scandium surrogate. Scandium must respond within 80-120% for all QC samples and within 60-120% for all other samples. See section 11.7 for further guidance.
- 10.15 Initial Calibration Blank (ICB)/Continuing Calibration Blank (CCB) - A calibration blank (either initial or continuing) is a volume of reagent water that is matrix matched to the calibration standards and is analyzed following each initial (ICV) and continuing calibration verification (CCV) standard. The ICB/CCB must not contain analytes $>\frac{1}{2}$ RL or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. If target analytes are present in the ICB/CCB, corrective action must be taken. See section 11.5 for corrective actions.
- 10.16 Sample Dilution - If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte Linear Dynamic Range (LDR), the sample must be diluted with acidified reagent water and re-analyzed.
- 10.17 Supporting Analytical Studies - Analysis of Instrument Detection Limits (IDL) are done every quarter and Linear Range Studies are completed every 12 months, IEC's are done every 6 months or sooner as needed, and MDL studies are done once a year.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 Initial Calibration - After analyzing the calibration standards, the curve is reviewed to ensure the acceptance criteria described in section 10.4 are met. If analytes do not meet this requirement, corrective action must be taken. Corrective actions may include re-calibrating the instrument, replacing the tubing on the peristaltic pump, examining the standards for degradation, or performing instrument maintenance. If the internal standard responses in the calibration standards do not meet the criteria in section 10.13, re-calibrate the instrument.

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- 11.2 Initial Calibration Verification (ICV/ICVLL) - Following acceptable calibration, the EPA method 6010B and 200.7 curves are verified by analyzing a second source standard (ICV) containing all target analytes. The EPA method 6010C curve is verified by analyzing a second source standard (ICV and ICVLL) containing all target analytes. If the criteria described in section 10.5 are not met for a target analyte, re-analyze the ICV/ICVLL. If this fails a second time, corrective action must be taken. Re-calibrate and re-analyze the ICV/ICVLL using the same standard. If acceptance criteria are still not met, re-check standard curve and ICV/ICVLL preparation and/or perform routine instrument maintenance. If still not acceptable, refer to manufacturer's instruction or call service representative.
- 11.3 Continuing Calibration Verification (CCV/CCVLL) - The results of the continuing calibration verification check standard:

For SW-846 Method 6010B - The continuing calibration verification standard must agree with the criteria in section 10.6 or the CCV must be re-analyzed. If the recovery fails a second time, corrective action must be taken. The corrective action would be to re-calibrate and re-analyze the last 10 samples, using the same CCV standard. If acceptance criteria are still not met, re-check the standard curve and CCV preparation and/or perform instrument maintenance. If the CCV still does not pass, refer to the manufacturer's instruction or call a service representative.

For SW-846 Method 6010C - The continuing calibration verification standard must agree with the criteria in section 10.6 or the CCV/CCVLL must be re-analyzed. If the recovery fails a second time, corrective action must be taken. The corrective action would be to re-calibrate and re-analyze the last 10 samples, using the same CCV/CCVLL standard. If acceptance criteria are still not met, re-check the standard curve and CCV/CCVLL preparation and/or perform instrument maintenance. If the CCV/CCVLL still does not pass, refer to the manufacturer's instruction or call a service representative.

For EPA Method 200.7 – The continuing calibration verification standard analyzed must agree with the criteria in section 10.6 or the CCV must be reanalyzed. If the recovery fails a second time, corrective action must be taken. The corrective action could be to re-calibrate and re-analyze the last 10 samples, using the same CCV standard. If acceptance criteria are still not met, re-check the standard curve and CCV preparation and/or perform instrument maintenance. If still does not pass, refer to the manufacturer's instruction or call a service representative.

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7300, 7301, AND 7303 AND OSHA ID-125G)**

- 11.4 Interference Check Standards (ICSA/ICSAB) - Evaluate the ICSA and ICSAB. The analyst must verify that the ICS and ICSAB have been analyzed at the required frequency. The prepared solutions are analyzed at the beginning and the end of each 8-hour shift. Results must meet the criteria described in section 10.10. If these criteria are not met, check the background correction protocols currently in place for appropriateness. If this is the initial ICS and/or ICSAB run after daily calibration, re-calibrate and re-analyze. If the ICSA and/or ICSAB did not agree at the end of an 8-hour shift, re-adjust background correction factors and, if needed, re-analyze any samples in the previous run that may have been affected.
- 11.5 Blanks - Evaluate the blanks. The analyst must confirm that both the method blanks and the continuing calibration blanks were analyzed at the required frequency. Other items to check are as follows:
 - 11.5.1 The instrument blank or continuing calibration blank (ICB/CCB) must meet the criteria in section 10.6. Common laboratory contaminant concentration may be present greater than half the RL, but must not be greater than the RL. If a target analyte value is above the RL for a common laboratory contaminant, consult Supervisor for proper corrective action. Complete a Corrective Action Report, if appropriate. Corrective actions may include re-prepping the samples, the site specific DQO's may allow the use of an elevated detection limit, or use of a "B" qualifier.
 - 11.5.2 Corrective actions for method blank contamination include re-prepping the entire batch of samples, or if the site-specific requirements can be met, an elevated detection limit may be used, or the sample data qualified with a "B" qualifier and footnote.
- 11.6 During the analysis of samples, the instrument supplies triplicate readings. The responses must have a %RSD of <20% for all results greater than the reporting limit. If any sample or QC fails to meet the 20% criteria, it must be evaluated for interferences. If interference is determined to be the cause, dilute the sample by 2X and re-analyze. Perform higher dilutions, if necessary. Instrument QC cannot fail the stated criteria. If failure occurs, the batch is invalid.
- 11.7 Surrogate - Evaluate scandium for presence/absence in each digestate. Scandium is added to all samples (prior to digestion) to ensure that the sample was prepped and that the instrument injected the sample. If scandium is not present in the sample, re-prep and re-analyze.

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- 11.8 Internal Standard - The intensity of the Yttrium internal standard response in each sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in each sample must meet the criteria in section 10.13. If the %RI of the response in the sample falls outside of these limits, the laboratory must immediately re-analyze the calibration blank and monitor the internal standard intensities. If the %RI for that calibration blank is within the limits, the laboratory must re-analyze the original sample at a two-fold dilution. If the %RI for the re-analyzed calibration blank is outside the limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, the new calibration verified, and the samples reanalyzed.
- 11.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - Assess that matrix spike/matrix spike duplicates were analyzed at the required frequency. MS/MSD recovery ranges are as follows

Method	Acceptance Criteria	
	Water	Soil
6010B & 6010C	75 – 125%	75 – 125%
200.7	75 – 125%	NA

If the MS/MSD recoveries are unacceptable, the analyst notifies the supervisor for corrective action. Corrective actions may include re-analysis, re-calculating results, and/or re-prepping the samples. If the MS/MSD recovery is outside the control limits and the laboratory control standard was within method control limits, then the recovery problem is judged to be matrix related. The LCS within method control limits demonstrates the laboratory performance was in control without the effect of sample matrix interference. The project manager must be contacted to discuss further alternatives and the potential impact on the project. Note: Only reanalyze at a dilution if a matrix interference is observed.

If the sample concentration for an analyte is greater than 4 times the spike concentration, a "V" qualifier is used. The "V" qualifier indicates that the high concentration of analyte in the sample interfered with the ability to make an accurate spike recovery determination.

Assess that matrix duplicates were analyzed at required frequency. Acceptance criteria are that all RPD results meet project-established goals. If no project specific goals are present for the sample batch, the results must be within 20% RPD. If these conditions are not met, perform the following corrective action as appropriate. Corrective actions may include re-analysis, re-calculating results, and/or re-prepping the samples.

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7300, 7301, AND 7303 AND OSHA ID-125G)

- 11.11 For each new batch, ensure that the following series of tests were performed and meet the listed acceptance criteria.

11.11.1 Serial Dilution: If the analyte concentration is sufficiently high, an analysis of a 1:4 dilution must agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.

11.11.2 Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, must be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect is suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

- 11.12 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - Assess that LCSs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.9, the affected samples must be re-digested and re-analyzed along with a new LCS. LCSD is only prepared and analyzed when the client does not submit sufficient sample volume for a duplicate, Matrix spike and Matrix Spike Duplicate. The accuracy requirement for the LCSD is the same as the LCS. If an LCSD was prepared and analyzed and fails to meet the acceptance criteria, rinse the instrument and re-analyze. If the LCSD fails for a second time, re-prepared all samples prepared in conjunction with the failing LCSD. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction.

If insufficient sample is available to re-digest all the samples, then a Corrective Action Report must be initiated.

- 11.13 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the MDL and below 90% of the LDR. Sample concentrations that have been analyzed using the extracts in its most concentrated form, and are below the Reporting Limit and is reported as <RL. Sample results that are above the MDL but below the reporting limits need to be flagged as estimates. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

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11.14 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method.

11.14.1 The analyst should review any sample that has quantifiable compounds.

11.14.2 All calculations must be checked.

11.14.3 Blanks must be clean of all interfering peaks.

11.14.4 Quality control criteria should be checked for the LCS, LCSD, MS, and MSD.

11.14.5 Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.14.6 See SOP# 030201, *Data Handling and Reporting*.

11.14.7 See SOP# 030208, *Corrective Action*.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan*.

12.2 See SOP #030302, *Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) listed.

13.2 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.3 If a modification to the procedure is required to analyze a sample beyond those specified in the procedure as state notes, it must be properly documented. Any modification to the method is listed in the test report's case narrative, and the analytical method is reported as modified.

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14.0 REFERENCES

- 14.1 EPA Method 6010B, Test Methods for the Analysis of Solid Wastes, Physical/Chemical Methods, Update III
- 14.2 EPA Method 200.7, Methods for the Determination of Metals in Environmental Samples, Rev. 4.4, May 1994
- 14.3 Code of Federal Regulations Title 40, Part 136.3
- 14.4 Code of Federal Regulations Title 40, Part 141.23
- 14.5 EPA Method 6010C, Test Methods for the Analysis of Solid Wastes, Physical/Chemical Methods, Rev. 3, February, 2007.
- 14.6 SM 2340B, Standard Methods, 20th Edition.
- 14.7 Method 7300, Elements by ICP, NIOSH Manual of Analytical Methods, 4th Edition
- 14.8 OSHA Method ID-215G, Metal and Metalloid Particulates in Workplace Atmospheres (ICP Analysis), Sampling and Analytical Methods, September 2002.
- 14.9 Method 7301, Elements by ICP, NIOSH Manual of Analytical Methods, 4th Edition
- 14.10 Method 7303, Elements by ICP, NIOSH Manual of Analytical Methods, 4th Edition

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
15	4/15/11	Technical and Quality Review and update. Added state notes where applicable; Added Tables 1.2b & 1.2c; Revised Table 1.2a and Sections 1.1, 1.3, 1.6, 1.11, 2.18, 2.22, 5.6, 7.1, 7.6.2, 7.9, 8.1.3, 8.1.5, through 8.1.10, 9.1, 9.7 through 9.12, 10.3, 10.0 & 11.0, 12.1; Added Sections 2.13.1, 2.14.1, 2.31 through 2.35, 3.1.1, 4.1, 4.7, 7.17, 13.2, 13.3, 14.5 through 14.10.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	5/1/95	Origination
1	7/25/95	
2	3/11/97	
3	8/18/99	
4	2/11/00	
5	8/21/00	
6	3/28/01	
7	12/14/01	
8	4/11/03	
9	1/26/04	
10	8/2/04	
11	10/15/05	Corrected CCV criteria for EPA 200.7
12	10/29/08	Technical and Quality Review and update. Corrected acceptance criteria in Section 10.6. Updated format and re-organized sections 8.0, 10.0 and 11.0 based on new format.
13	1/23/09	Technical and Quality Review and update.
14	2/2/09	Technical and Quality Review and update. Clarification of holding times, Inclusion of cross-references. Inclusion of section 13.1 and section 7.1.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

SOP NUMBER: 340384B

Prepared by: Ryan Holdbrooks/Jim Burns

Reviewed by: Jim Burns/Dixie Marlin

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Department Manager

QA Department

1.0 SCOPE AND APPLICATION

- 1.1 This cold vapor atomic absorption procedure is for determining the concentration of mercury in solids, sediments and sludge. The routine laboratory reporting limit is 0.4 μ g/kg; however reporting limits are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.
- 1.2 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.

STATE NOTE: For samples regulated under the Ohio VAP, this SOP is approved only for soil/sediment/sludge samples.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The goals of the Mercury system are to convert all Hg species to Hg⁺⁺ ions and break down all organic molecules in each sample. Organic molecules must be broken down to prevent their interference with mercury sample analysis. By adding an appropriate sequence of reagents to each sample and heating in a hot block for 30 minutes, all organic molecules are broken down and all Hg species are completely oxidized to Hg⁺⁺ ions, used for cold vapor atomic absorption analysis.
- 2.2 A soil sample digestate with mercury in the divalent form enters the system and is mixed with a reducing agent (SnCl₂) to form elemental mercury vapor.

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- 2.3 The dry vapor then enters the optical cell that has been optimized for fast response time and sensitivity. A mercury light source emits a stable source of light at 253.7nm wavelength. The intensity of the light source passing through the mercury cold vapor cell is measured using a solid-state detector with a wide dynamic range. To measure the mercury concentration, the resulting sample absorbance signal is compared to the absorbance of the pure carrier gas flowing through the optical path under identical conditions.
- 2.4 Calibration Standard (CAL) – Solutions of known concentrations used to create graphic representation of the relationship between the known values, such as concentrations and instrument responses.
- 2.5 Instrument Detection Limit (IDL) - The smallest signal above background noise that an instrument can reliably detect.
- 2.6 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be measured with 99% confidence that the analyte concentration is greater than zero.
- 2.7 Linear Dynamic Range (LDR) – The concentration range where absorbance and concentration remain directly proportional to each other. A wide linear dynamic range permits the analysis of a wide range of sample concentrations (optical densities) and reduces sample preparation (dilution) requirements.
- 2.8 Batch - A group of samples (not exceeding 20 total) having the same matrix and analyzed at the same time using the same conditions and procedures.
- 2.9 Method Blank - An analytical control consisting of all reagents that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.
- 2.10 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) – Duplicate aliquots of a control sample of known composition. This sample is prepared from a source that is different from the stock used to prepare the initial and continuing calibration standards. LCS/LCSD are analyzed exactly like a sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Method precision can be determined using the results of the LCS/LCSD analysis.
- 2.11 Duplicate – A second aliquot of a field sample analyzed using identical preparation and analytical procedures. Analysis of a sample duplicate monitors precision associated with laboratory procedures.

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- 2.12 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine the appropriateness of the method for the field matrix by measuring recovery of target analytes. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.
- 2.13 Reagent Water – Water in which an interferent is not observed at or above a specified concentration for the parameter of interest.
- 2.14 Initial Calibration Verification (ICV) - Analytical standard analyzed after the initial calibration to verify the initial calibration of the system. The ICV is prepared using a different source than is used to prepare the standards for the initial calibration. A Low Level ICV (ICVLL) can be a same or separate source from the calibration standards and is analyzed following the calibration curve to verify the initial calibration of the system at the reporting limit.
- 2.15 Continuing Calibration Verification (CCV) - Analytical standard run after every 10 samples and at the conclusion of the analytical sequence to confirm that the instrument maintains calibration stability within acceptable limits. The CCV is prepared from the same source as the standards used in the initial calibration. The CCV may not be used to replace the ICV. A Low Level CCV (CCVLL) is analyzed at the same frequency as the CCV to verify the continued stability of the analytical system at the reporting limit.
- 2.16 Initial Calibration Blank (ICB) – A blank that is analyzed immediately after ICV. The ICB is a contamination and memory effect check solution.
- 2.17 Continuing Calibration Blank (CCB) - A blank that is analyzed immediately after every CCV. The CCB is a contamination and memory effect check solution.
- 2.18 Serial Dilution (SD) – A subsequent dilution of a high concentration field sample that should agree within acceptance criteria of the original undiluted analysis. This is generally used as a test for matrix interferences or matrix effects.
- 2.19 Post Spike (PS) – A standard prepared from a previously analyzed spiked sample digestate that yielded reduced recovery for the target analyte due to a suspected matrix interferent.

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3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets are also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.
- 3.2 Use of this procedure requires handling acid preserved samples, standards and concentrated acids. Always wear safety glasses, protective gloves and laboratory coat. Also, fume hoods must be used for both the automated and manual preparation of mercury samples. Mercury vapor is toxic: precaution must be taken to avoid inhalation.
- 3.3 Before preparing any samples, analyst must be familiar with all safety techniques involving strong acids and any of the other chemicals or reagents used. Refer to the appropriate Material Safety Data Sheets (MSDS) for all pertinent information.
- 3.4 Before starting a sample preparation run, check to ensure that the vent hood connection is drawing at the required flow rate. The exhaust may contain minute amounts of mercury or other potentially dangerous chemicals.
- 3.5 Many mercury-containing compounds are highly toxic, if swallowed, inhaled or absorbed through the skin. Extreme care must be exercised in the handling of concentrated mercury reagents. These reagents should only be handled by analysts knowledgeable of their risks and of safe handling procedures.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.
- 4.2 Soil samples are stored at $4^{\circ}\pm 2^{\circ}\text{C}$.
- 4.3 Holding times for field samples are 28 days from sample collection to preparation.
- 4.4 All glassware must be washed with Alconox, tap water rinsed, nitric acid rinsed and then rinsed with DI water. See ESC SOP# 030701, *Glassware Cleaning*.
- 4.5 Samples can be collected in either plastic or glass containers.

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5.0 INTERFERENCES

- 5.1 Potassium permanganate is added to eliminate possible interference from sulfide. The KMnO₄ oxidizes the sulfides.
- 5.2 It has been reported that copper could cause interference though the laboratory has not detected this effect.
- 5.3 Chlorides can also cause interferences; so additional potassium permanganate is added to oxidize chloride to free chlorine. Free chlorine absorbs radiation at 254nm, so an excess of hydroxylamine sulfate is added to remove free chlorine.
- 5.4 Some volatile organic materials that absorb at 253.7nm could cause interference. If interference is a problem during a sample run, a preliminary run without reagents determine if this type of interference is present.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Perkin Elmer FIMS400 Mercury Analyzer, or equivalent. (Instruments #1, #2, #3)
- 6.2 Polypropylene culture test tubes, 15mL and 50mL capacity.
- 6.3 10 mL graduated glass disposable serological pipettes. (Pyrex or equivalent)
- 6.4 Class A 100mL volumetric flasks (Pyrex or equivalent).
- 6.5 Class A volumetric pipettes, 1mL and 5mL.
- 6.6 8 cup capacity standards rack
- 6.7 98 cup capacity sample rack
- 6.8 60mL disposable polypropylene sample containers
- 6.9 Mettler analytical balance, or equivalent (Serial number 1114501486, or equivalent).
- 6.10 Bulk Liquid Compressed Argon gas, pre-purified, used as instrument carrier gas.
- 6.11 Precision Hot Block (Brand name - MOD Blocks, or equivalent).
- 6.12 Computer software used: (AA WINLAB, version 2.50, or equivalent).
- 6.13 Sample introduction system (auto sampler)- (Model, instrument #1 & #3 is a Perkin Elmer AS91, and for instrument #2 a Perkin Elmer AS90, or equivalent).

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- 6.14 Peristaltic pump, to pump reagents and samples through the detector.
- 6.15 Computer used is a COMPAQ, or equivalent.
- 6.16 Adjustable-volume pipetter. (Eppendorf, or equivalent)

7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner if a problem is detected, unless otherwise noted.
- 7.2 ASTM Type 1 water - Water must be free of mercury or anything that may interfere with the mercury analysis.
- 7.3 Stock standards expiration date is one year from the date received or the expiration date assigned by the manufacturer whichever is sooner.
- 7.4 Concentrated hydrochloric acid (HCl) – Concentrated, trace metal grade (OMNI TRACE, catalog# HX0607-2, or equivalent). NOTE: manufacturer shelf life/expiration date.
- 7.5 Concentrated nitric acid (HNO₃) – Concentrated, trace metal grade (VWR EM-Nx0407-2 or equivalent). NOTE: manufacturer shelf life/expiration date.
- 7.6 5% Potassium Permanganate (KMnO₄) solution – JT Baker 3227-01, or equivalent. Weigh 500 ± 0.01g of potassium permanganate per 10L reagent water.
- 7.7 Sodium Chloride-Hydroxylamine Sulfate – prepared by placing 240 ± 0.01g of Hydroxylamine sulfate, JT Baker N646-07, or equivalent, and 240 ± 0.01g of sodium chloride, EM Science SX0420-1, or equivalent, into 2L reagent water.
- 7.8 1.1% Stannous Chloride – JT Baker 3980-11, or equivalent. Weigh 22.0 ± 0.01g SnCl₂ and 60mL conc. HCl (3%) in a 2L volumetric flask and dilute to 2L with DI water. **Made fresh DAILY.**
- 7.9 Aqua Regia – Prepare immediately before use, by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃.

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- 7.10 Primary Stock Mercury Solution: 1000ppm, Ultra Scientific, Cat # ICP-080, or equivalent. This primary stock solution is used for calibration standards, continuing calibration verification (CCV), Low Level Initial Calibration Verification (ICVLL), Low Level Continuing Calibration Verification (CCVLL), Matrix Spike (MS), and Matrix Spike Duplicate (MSD). See section 13.4.
- 7.11 Secondary Source Stock Mercury Solution: 1000ppm, Inorganic Ventures, cat# AAHG1-5, or equivalent. This stock solution is used for initial calibration verification (ICV). See section 13.4.
- 7.12 3PPM Intermediary Standards: Make up this concentration from both sources, the Primary 1000PPM source from Ultra Scientific, and the Secondary 1000PPM source from Inorganic Ventures. For the 3PPM Primary intermediate solution, spike 0.3mL of the 1000PPM Ultra Scientific stock solution into 100mL Class A volumetric flask with approximately 50mL of DI water and 10mL of concentrated nitric acid and bring up to volume with DI water. For the 3PPM Secondary Intermediate solution, spike 0.3mL of the 1000PPM Inorganic Ventures stock solution into 100mL Class A volumetric flask with approximately 50mL of DI water and 10mL of concentrated nitric acid and bring up to volume with DI water. These intermediate standards are made up fresh once a month. The 3PPM solution from Inorganic Ventures, is used for the ICV. See section 13.4.
- 7.13 300PPB Intermediary Standards.
Make up this concentration the Intermediary 3PPM source from Ultra Scientific. For the 300PPB intermediate solution, spike 5.0mL of the 3PPM Ultra Scientific stock solution into 50mL Class A volumetric flask with approximately 40mL of DI water and 5mL of concentrated nitric acid and bring up to volume with DI water. This intermediate standard is made fresh weekly. See section 13.4.

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8.0 PROCEDURE

8.1 Digestion

- 8.1.1 For the calibration curve and instrument QC (ICVs and CCVs), using a continuously adjustable-volume pipetter, add 5mL of DI water to the digestion tubes and spike each level with the appropriate amount of intermediate standard, and continue prepping per method. Typical spiking levels are as follows; however see Section 13.4 for more information:
 - 0.0ppb - No spike
 - 0.2ppb - 20uL of 300ppb Ultra Scientific Intermediate
 - 0.4ppb - 40uL of 300ppb Ultra Scientific Intermediate
 - 1.0ppb - 100uL of 300ppb Ultra Scientific Intermediate
 - 2.0ppb - 200uL of 300ppb Ultra Scientific Intermediate
 - 5.0ppb - 50uL of 3ppm Ultra Scientific Intermediate
 - 10.0ppb - 100uL of 3ppm Ultra Scientific Intermediate
 - ICV-3.0ppb - 30uL of 3ppm Inorganic Ventures Intermediate
 - CCV-2.5ppb - 25uL of 3ppm Ultra Scientific Intermediate
 - ICVLL-0.4ppb - 40uL of 300ppb Ultra Scientific Intermediate
 - CCVLL-0.4ppb - 40uL of 300ppb Ultra Scientific Intermediate
- 8.1.2 For the method blank, weigh out 0.6 ± 0.5 g of JT Baker sand into the digestion tube. Using adjustable volume pipetter, add 5mL of DI water to the digestion tube. Aliquots of this blank will serve as the method blank, the ICB, and the CCB during analysis.
- 8.1.3 For the LCS, weigh out 0.6 ± 0.5 g of Environmental Resource Associates (ERA) into the digestion tube, using adjustable volume pipetter, add 5mL of DI water to the digestion tube. This weight may vary, depending on the concentration of the Hg standard from one lot# to the next.
- 8.1.4 For the samples, weigh out 0.6 ± 0.5 g of each field sample and record the weight. Prior to weighing, mix the sample to ensure that it is homogenized. If there are large solid artifacts such as stones and concrete, these can be broken up with a mortar and pestle before the sample is weighed. Note the final weight in the prep log. With the adjustable volume pipetter, add 5mL of DI water into each of the digestive tubes.
- 8.1.5 For the MS/MSD, weigh out 0.6 ± 0.5 g of a specified client sample, or a randomly selected client sample if none are specified, into the digestive tube. Using adjustable volume pipetter, add 5mL of DI water to the digestive tube. Then spike 50uL of the 3ppm Ultra Scientific Intermediate standard into the digestive tube.

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- 8.1.6 Add 2.5mL of Aqua Regia to all samples, standards and QC samples and cap tightly.
- 8.1.7 Heat for two minutes in a hot block at $95 \pm 3^\circ\text{C}$.
- 8.1.8 Cool for a minimum of five minutes. Add 25mL of DI water, swirl to mix, then add 5.0mL of 5% potassium permanganate solution. Swirl to mix. Allow purple color to persist for at least fifteen minutes. If the purple color persists move to step 8.1.9. If the purple color does not persist, add additional amounts of potassium permanganate crystals until the sample stays purple.
- 8.1.9 Heat in a hot block for at least thirty minutes at $95 \pm 3^\circ\text{C}$. Record the time-in and temperature-in of the hot block, on the Hg bench sheet.
- 8.1.10 After thirty minutes of digestion, take the sample out of the hot block and allow them to cool for a minimum of ten minutes. Record the time-out and temperature-out of the hot block. If any sample is colorless, it must be re-digested since the sample may have been lost due to insufficient amount of potassium permanganate added prior to digestion, or due to the matrix of the sample. If the temperature-out is outside the $95 \pm 3^\circ\text{C}$ range, the samples must be re-digested.
- 8.1.11 After cooling, add 2mL of sodium chloride-hydroxylamine sulfate to reduce the excess potassium permanganate. Swirl until purple color is gone. If the purple color persists, extra hydroxylamine sulfate crystals can be added. Allow sample to cool further, approximately 10-15 minutes, or to room temperature, and then adjust to final volume with DI water, as needed, using the graduations on the digestion vessel that are certified volumetrically by the manufacturer. The samples are ready for analysis. The samples are ready to analyze.
- 8.1.12 Stannous Chloride-HCl (SnCl_2) mix is not added during the digestion process, but is added during analysis through the instrument peristaltic pump. It is mixed with each sample, QC sample and all standards during the analysis.
- 8.2 Mercury Calibration and Analysis:
 - 8.2.1 Allow the detector to warm up at least thirty minutes before analyzing samples.
 - 8.2.2 Turn on the auto sampler and pump. Clamp pump tubing down. Make sure the argon gas is turned on and that argon gas flow is present. Ensure that the SnCl_2 and DI water are flowing. If the pump is not running correctly, re-adjust the tubing on the pump.
 - 8.2.3 Turn on the FIAS Mercury unit and prime the instrument with reagents for thirty minutes.

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- 8.2.4 Enter in the computer, the sample run (standards, QC samples and then the samples).
- 8.2.5 After all the information for the sample run has been entered in the computer, and the instrument has been primed, load the digested calibration standards, QC samples, and then the samples into the auto sampler rack. The peristaltic pump mixes the $\text{SnCl}_2\text{-HCl}$ solution into the field samples and transfers the standards and samples into the analyzer. Begin the analytical sequence.
- 8.3 Method performance criteria and corrective action procedures are found in Sections 10 & 11 of this procedure.

9.0 DATA ANALYSIS AND CALCULATIONS

- 9.1 Linear calibration model:

$$y = mx + b$$

where:
 y = Response A_x for External Standard
 x = Concentration C_x for External Standard
 m = Slope
 b = Intercept

Slope (m):

$$m = \frac{[(Swx_j y_j * Sw) - (Swx_j * Swy_j)]}{[(Sw * Swx_j^2) - (Swx_j * Swx_j)]}$$

Intercept (b):

$$b = y_{AVE} - (m * (x_{AVE}))$$

Correlation Coefficient (r):

$$r = \frac{[(Sw * Swx_j y_j) - (Swx_j * Swy_j)]}{\sqrt{[(Sw * Swx_j^2) - (Swx_j * Swx_j)] * [(Sw * Swy_j^2) - (Swy_j * Swy_j)]}}$$

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Coefficient of Determination (r^2):

$$r^2 = r * r$$

Where: n = number of x, y pairs

x_i = individual values for the independent variable

y_i = individual values for the dependent variable

w = weighting factor, for equal or no weighting w = 1

x_{AVE} = average of the x values

y_{AVE} = average of the y values

S = the sum of all the individual values

- 9.2 For total mercury in solid samples, round the solution analyte concentrations (mg/L) as instructed in Section 10.14. Report the data up to two significant figures as mg/kg dry-weight basis, unless specified otherwise by the program or data user. Calculate the concentration using the equation:

$$\text{Sample Conc. (mg/kg)} = \frac{C \times V \times D}{W}$$

where: C = Concentration in extract (mg/L)

V = Volume of extract (L, 100 mL = 0.1L)

D = Dilution factor (undiluted = 1)

W = Weight of sample aliquot extracted (9 g x 0.001 = kg)

- 9.3 Soil samples are reported on a dry weight basis. After a dry weight for each sample has been obtained, the calculations are performed as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100$$

where: DW = Sample weight (g) dried

WW = Sample weight (g) before drying

At ESC, dry weigh conversions are performed automatically by the LIMS data system.

- 9.4 LCS/ICV/CCV Percent Recovery (%R):

$$\% R = \frac{\text{Measured concentration}}{\text{Actual concentration}} \times 100$$

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9.5 Matrix Spike Recoveries (%R_{MS/MSD}):

$$\%R_{MS/MSD} = \frac{O_i - O_s}{T_i} \times 100$$

where: O_i = observed sample concentration with the spike added
O_s = the observed value for the sample without the spike
T_i = True value of the spike added

9.6 Relative Percent Difference (%RPD):

$$RPD = \frac{\frac{Value~1 - Value~2}{Value~1 + Value~2}}{2} \times 100$$

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.
- 10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP 030201, *Data Handling and Reporting*.

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10.3 Batches:

Preparation batches are defined as sets of 1 – 20 samples. Preparation batch analysis must include the following: 1 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), if sufficient volume is not available for a Sample Duplicate, 1 Sample Duplicate (DUP), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information must be maintained in the preparation documentation assigned to the department.

Analytical batches require all the samples contained within the previous preparation batches and additional instrument operating requirements. Analytical batches include: 1 Initial Calibration Verification (ICV) sample, 1 Low Level Initial Calibration Verification (ICVLL) sample, 1 Initial Calibration Blank (ICB), 1 Low Level Continuing Calibration Verification (CCVLL) sample following every 10th sample and at the conclusion of the sequence, 1 Continuing Calibration Verification (CCV) sample following every 10th sample and at the conclusion of the sequence, 1 Continuing Calibration Blank following each CCV, 1 Serial Dilution, if possible, and 1 Post Spike.

- 10.4 Initial Calibration - The curve is prepared daily and must consist of at least five standards and a blank. The calibration is acceptable when the correlation coefficient is \geq 0.998. The concentration of the curve analyzed are as follows, 0.0, 0.2, 0.4, 1.0, 2.0, 5.0, and 10.0 ppb. See section 13.4. The calibration curve must contain a standard at or below the reporting limit. The blank is included as a point in the calibration curve to account for any background interferences that may be present in the digestion solutions. Do not force the curve regression fit through zero.
- 10.5 Initial Calibration Verification (ICV)/Low Level Initial Calibration Verification (ICVLL) - After the passing standardization is achieved, analyze the low level initial calibration verification standard (ICVLL). The ICVLL recovery must be within $\pm 30\%$ of the true value. Following the ICVLL, the mid-range ICV is analyzed. The mid-range ICV must be within $\pm 10\%$ of the true value.
- 10.6 Continuing Calibration Verification (CCV) - After every ten samples and at the end of the analysis, analyze the low level continuing calibration verification standard (CCVLL). The CCVLL recovery must be within $\pm 30\%$ of the true value. Following the CCVLL, the mid-range CCV is analyzed. The mid-range CCV must be within $\pm 10\%$ of the true value.
- 10.7 Method Blank/Initial & Continuing Calibration Blank (ICB/CCB) - One method blank must be analyzed for every twenty samples. The ICB is analyzed following every Initial Calibration Verification (ICV) standard and the CCB is analyzed following each CCV and at the conclusion of the sequence. Mercury should not be detected in the blank $>\frac{1}{2}$ reporting limit (RL).

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, the method blank/ICB/CCB concentration must be less than the reporting limit.

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- 10.8 Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD) – An LCS must be performed with each batch of twenty field samples. An LCSD is also prepared if there is insufficient field sample volume for a Sample Duplicate. The LCS/LCSD recoveries must be within the performance acceptance limits provided by the standard vendor (ERA). The concentration and true value is subject to change from one lot# to another.
- 10.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - An MS & MSD must be analyzed with every batch of samples. The MS & MSD are prepared by spiking two separate aliquots of a field sample with a known amount of standard. The MS and MSD are prepped and analyzed in the same manner as the samples. Recovery must be within $\pm 20\%$ of the true value for accuracy and the relative percent difference (RPD) must not exceed $<20\%$.
- 10.10 Sample Duplicate (DUP) – If sufficient sample volume is submitted by the client, one sample duplicate is analyzed for every twenty samples. A sample duplicate is a second aliquot of a field sample that has been taken through the entire procedure. The RPD of the parent sample and the duplicate must be $<20\%$.
- 10.11 Serial Dilution - A serial dilution must be run with each batch of samples analyzed. A serial dilution is analyzed if a sample is $x25$ the MDL or higher. A serial dilution is, at a minimum, a 5 times dilution of the sample, and must agree within 10% of the original value.
- 10.12 Post Spike - If the MS/MSD recovery fails, or if an MS/MSD is not analyzed, due to insufficient sample, a post spike must be analyzed when the sample concentration is less than $x25$ the MDL. If the sample concentration is less than the MDL, spike the sample at $x20$ the MDL. The criteria for the post spike must be within $\pm 15\%$ of the true value. **If the MS/MSD recovery passes a post spike need not be analyzed.**
- 10.13 Dilutions - Any sample, with a concentration over the high standard in the curve, must be diluted within the range of the calibration. This dilution must be at the lowest dilution possible, to keep reporting limits as low as possible.
- 10.14 Digestion Temperature - The hot block's temperature is monitored using a temperature blank containing a thermometer that is calibrated against a NIST traceable reference thermometer. The temperature blank is moved to a different position in the hot block each time samples are digested. The daily temperature blank is documented each day in the appropriate logbook.

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- 10.15 Sample Analysis - During the analysis of the samples, the instrument supplies triplicate readings. The responses must have a %RSD of <20% for all results >RL. The instrument automatically reports the mean of triplicate scans using a simple average calculation: A+B+C/3 = reported concentration for the sample.

Do not report data below the determined analyte reporting limit concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required by the analysis.

Sample data should be reported in units of mg/Kg for soil samples on a dry weight basis. For soil samples report the data generated directly from the instrument with allowance for any sample dilution.

When reporting, round the data values to the tenth place and report analyte concentrations up to two significant figures. Extract concentrations for solids data are rounded in a similar manner before calculations in Section 9.2 are performed.

- 10.16 Instrument Detection Limit (IDL) - The instrument detection limit is calculated by performing ten sequential replicate measurements of a method blank. The standard deviation of these measurements is calculated and the IDL is equal to three times the standard deviation of the measurements. The IDL assures with 99% certainty that a value is above the instrument noise level. This IDL study is done on a quarterly basis.

NOTE: An IDL is a statistical determination without analytes present and an MDL is determined with low levels of analytes present.

- 10.17 For corrective actions, see section 11.0.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

- 11.1 All sample data must undergo a QC review.

11.1.1 The reviewer must verify that all reportable results are derived from data that are within the calibration range.

11.1.2 Analysts signs and dates the appropriate raw data, printouts and bench sheets

11.1.3 All calculations must be checked.

11.1.4 Data must be checked to confirm that all required QC checks have been analyzed and that they are within acceptable limits.

11.1.5 Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

- 11.1.6 See SOP #030201, *Data Handling and Reporting*.
- 11.2 Initial Calibration – Since calibration curves are prepared and digested with field samples, any curve that is prepared and analyzed that does not meet the acceptance criteria must be re-prepared, along with all the relevant field samples and QC that are prepared in conjunction with the calibration curve.
- 11.3 ICV – If the first run of the mid-range ICV does not pass the $\pm 10\%$ criteria and/or the ICVLL does not pass the $\pm 30\%$ criteria, rinse and rerun the standard once. If this fails, corrective action must be taken. The corrective action includes re-calibration and re-analysis, using the same ICV/ICVLL standard. If acceptance criteria are still not met, re-check the standard curve and ICV/ICVLL preparation and/or perform instrument maintenance. If still does not pass, refer to manufacturer's instruction manual, or call a service representative. Since the standards are digested in conjunction with the field sample, if failures result from the standards, all samples within the preparation batch must be re-digested.
- 11.4 CCV – If the first run of the CCV or the CCVLL does not pass, it is re-analyzed once. If the failure continues, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze all samples not bracketed by acceptable CCVs/CCVLLs. After re-calibration, if the CCV/CCVLL still fails, the entire prep batch must be re-digested.
- 11.5 Blanks (Method/ICB/CCB) - If the Method Blank, ICB, or CCB fails the acceptance criteria, stop the run and re-analyze once. If the Method Blank fails in the second analysis, re-calibrate the instrument. After re-calibration, if the method blank still fails, all samples within the prep batch must be re-digested. If following re-analysis the ICB or CCB failure persists, re-calibrate and re-analyze the ICB/CCB and any field samples not associated with acceptably bracketing ICB/CCBs. If the failure persists following recalibration and re-analysis, the entire prep batch must be re-digested.
- 11.4 LCS/LCSD – If the LCS fails the criteria, stop the run and re-analyze one more time. If the LCS still fails, re-calibrate the instrument. After recalibration, if the LCS fails again, all samples within the prep batch must be re-digested. If an LCSD is performed, it must also meet the LCS acceptance criteria provided by the manufacturer.

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- 11.5 MS/MSD – If the MS/MSD fails the criteria, stop the sequence and re-analyze once. If the MS/MSD failure persists, run a post spike. The post spike must pass within the acceptance criteria listed in section 10.12. If the post fails and the LCS was within method limits, then the recovery problem with the MS/MSD is judged to matrix-related. If the LCS is within method control limits, this demonstrates the laboratory performance was in control in a clean matrix. Re-digestion may still be necessary however, if the supervisor or the client requests a re-digestion. If re-digestion is not performed, the failures must be flagged with a “J5” (the sample matrix interfered with the ability to make any accurate determination; spike value is high), or “J6” (the sample matrix interfered with the ability to make any accurate determination; spike value is low). If there is a RPD failure, a re-prep is be used to confirm results, when possible.
- 11.6 Sample Duplicate - If a duplicate is analyzed and the duplicate RPD is outside the control limits (>20%), results are flagged with a “J3” (the associated batch QC was outside the established quality control range for precision), re-digestion may be necessary. Consult your supervisor.
- 11.7 Serial Dilution – If the dilution does not meet the required acceptance criteria, redilute the sample and re-analyze. If the failure persists, re-analyze both the parent sample and the dilution. If failure still occurs, re-calibrate and re-analyze both the parent and the dilution.
- 11.8 Post Spike – If the MS/MSD recoveries pass the acceptance criteria, a post spike is not required. If performed and the post spike does not meet the acceptance criteria, re-analyze once. If the failure persists, re-analyze both the parent sample and the spike. If failure still occurs, dilute the parent sample and re-spike and re-analyze. If the re-analysis passes, the confirmation of a matrix effect is confirmed and must be noted on the final client report.
- 11.9 Sample Analysis - Verify that all triplicate responses are represented by a %RSD of <20%. Any result exhibiting a % RSD above 20% requires an initial dilution of 2X and subsequent dilutions as necessary. Apply a “J3” (the associated batch QC was outside the established quality control range for precision) qualifier to the sample to notify the end user of the interference and necessity for the dilution.
- 11.10 Digestion Temperature - If the temperature-out is outside $95 \pm 3^{\circ}\text{C}$ range after the thirty minutes of digestion, re-digest the entire batch.
- 11.11 Digestate - If any sample is colorless after digestion, the sample must be re-digested, since the sample may have been lost due to insufficient amount of potassium permanganate added prior to the digestion, or due to the matrix of the sample.

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- 11.12 Sample Concentrations - The analyst must verify all reported results are derived from the analytical results that are both above the MDL and below the high standard used in the curve.

Sample concentrations that have been analyzed using the extracts in its most concentrated form, and are <RL, report the result as <RL.

For sample results that are above the MDL, but below, the reporting limit, these results must be flagged as estimated values (J flag).

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. blank, spike, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *Waste Disposal*, ESC SOP # 030301

- 12.2 See SOP #030302, *Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

- 13.1 HCl is used in the SnCl₂ mixture per the instrument manufacturer's instruction manual that specifies HCl in the SnCl₂ instead of sulfuric acid.
- 13.2 The stannous chloride solution concentration has been modified in this procedure to reflect the instrument manufacturer's recommendation (Mercury Hydride Analysis Flow Injection (pg. 2/13)).
- 13.3 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) listed.
- 13.4 Adjustments to reporting limits, the concentrations of standards/spiking solutions, standards providers, and quality control recovery/RPD limits are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

14.0 REFERENCES

- 14.1 Method 7471A, Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique). Third Edition Rev.1 September 1994.
- 14.2 Perkin Elmer FIMS 400 Instruction Manual.
- 14.3 Method 7471B, Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique). Rev.2, February 2007.
- 14.4 Method 7000B, Flame Atomic Absorption Spectrophotometry, Rev.2, February 2007.
- 14.5 Method 7000A, Atomic Absorption Methods, Rev.1, July 1992.

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TITLE: MERCURY IN SOLID WASTE (COLD-VAPOR TECHNIQUE) (EPA METHODS 7471A & 7471B)

Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
5	9/20/10	Technical and Quality Review and update. Revised sections 1.1 and added state note, 2.4, 2.5, 2.7, 2.10, 2.13, 2.14, 2.15, 2.18, 2.19, 5.3, 7.1, 7.10, 7.11, 7.13, 8.1, 8.2, 9.1, 9.2, 9.3, 9.6 through 9.6, 10.3, 11.1 through 11.12, 12.1, Added sections 3.5, 10.4 through 10.17, 13.4, 14.3 through 14.5

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	7/30/04	Origination
1	12/5/05	
2	11/3/06	
3	1/23/09	Technical and Quality Review and update.
4	1/29/09	Clarification of holding time; Addition of sections 9.1, 9.2, State Notes, 12.1-12.2, & 13.3; Ohio VAP approval 1/29/09

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ATTACHMENT B

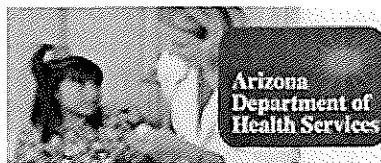
**ENVIRONMENTAL SCIENCE CORP. - LIST OF REPORTING LIMITS FOR
EPA METHOD 8260B**

VOCs 8260 GW (updated 6-28-07) MS				
Cas#	Parameter	Method	RDL	MDL
67-64-1	Acetone	8260B	0.05 mg/l	0.017 mg/l
107-02-8	Acrolein	8260B	0.05 mg/l	0.013425 mg/l
107-13-1	Acrylonitrile	8260B	0.01 mg/l	0.001457 mg/l
71-43-2	Benzene	8260B	0.001 mg/l	0.000312 mg/l
108-86-1	Bromobenzene	8260B	0.001 mg/l	0.000447 mg/l
75-27-4	Bromodichloromethane	8260B	0.001 mg/l	0.000371 mg/l
75-25-2	Bromoform	8260B	0.001 mg/l	0.000433 mg/l
74-83-9	Bromomethane	8260B	0.005 mg/l	0.000889 mg/l
104-51-8	n-Butylbenzene	8260B	0.001 mg/l	0.000245 mg/l
135-98-8	sec-Butylbenzene	8260B	0.001 mg/l	0.000281 mg/l
98-06-6	tert-Butylbenzene	8260B	0.001 mg/l	0.0003 mg/l
56-23-5	Carbon tetrachloride	8260B	0.001 mg/l	0.00036 mg/l
108-90-7	Chlorobenzene	8260B	0.001 mg/l	0.000307 mg/l
124-48-1	Chlorodibromomethane	8260B	0.001 mg/l	0.000308 mg/l
75-00-3	Chloroethane	8260B	0.005 mg/l	0.00046 mg/l
110-75-8	2-Chloroethyl vinyl ether	8260B	0.05 mg/l	0.005976 mg/l
67-66-3	Chloroform	8260B	0.005 mg/l	0.00054 mg/l
74-87-3	Chloromethane	8260B	0.0025 mg/l	0.000646 mg/l
95-49-8	2-Chlorotoluene	8260B	0.001 mg/l	0.00032 mg/l
106-43-4	4-Chlorotoluene	8260B	0.001 mg/l	0.000333 mg/l
96-12-8	1,2-Dibromo-3-Chloropropane	8260B	0.005 mg/l	0.000894 mg/l
106-93-4	1,2-Dibromoethane	8260B	0.001 mg/l	0.000363 mg/l
74-95-3	Dibromomethane	8260B	0.001 mg/l	0.000791 mg/l
95-50-1	1,2-Dichlorobenzene	8260B	0.001 mg/l	0.000303 mg/l
541-73-1	1,3-Dichlorobenzene	8260B	0.001 mg/l	0.000276 mg/l
106-46-7	1,4-Dichlorobenzene	8260B	0.001 mg/l	0.000301 mg/l
75-71-8	Dichlorodifluoromethane	8260B	0.005 mg/l	0.000403 mg/l
75-34-3	1,1-Dichloroethane	8260B	0.001 mg/l	0.000316 mg/l
107-06-2	1,2-Dichloroethane	8260B	0.001 mg/l	0.000342 mg/l
75-35-4	1,1-Dichloroethene	8260B	0.001 mg/l	0.000438 mg/l
156-59-2	cis-1,2-Dichloroethene	8260B	0.001 mg/l	0.000439 mg/l
156-60-5	trans-1,2-Dichloroethene	8260B	0.001 mg/l	0.000327 mg/l
78-87-5	1,2-Dichloropropane	8260B	0.001 mg/l	0.000535 mg/l
563-58-6	1,1-Dichloropropene	8260B	0.001 mg/l	0.000343 mg/l
142-28-9	1,3-Dichloropropane	8260B	0.001 mg/l	0.000347 mg/l
10061-01-5	cis-1,3-Dichloropropene	8260B	0.001 mg/l	0.000262 mg/l
10061-02-6	trans-1,3-Dichloropropene	8260B	0.001 mg/l	0.000319 mg/l
594-20-7	2,2-Dichloropropane	8260B	0.001 mg/l	0.000365 mg/l
108-20-3	Di-isopropyl ether	8260B	0.001 mg/l	0.000367 mg/l
100-41-4	Ethylbenzene	8260B	0.001 mg/l	0.000256 mg/l
87-68-3	Hexachlorobutadiene	8260B	0.001 mg/l	0.00058 mg/l
98-82-8	Isopropylbenzene (Cumene)	8260B	0.001 mg/l	0.000308 mg/l
99-87-6	p-Isopropyltoluene	8260B	0.001 mg/l	0.000286 mg/l
78-93-3	2-Butanone (MEK)	8260B	0.01 mg/l	0.002687 mg/l
75-09-2	Methylene Chloride	8260B	0.005 mg/l	0.000382 mg/l
108-10-1	4-Methyl-2-pentanone (MIBK)	8260B	0.01 mg/l	0.002 mg/l
1634-04-4	Methyl tert-butyl ether	8260B	0.001 mg/l	0.000316 mg/l
91-20-3	Naphthalene	8260B	0.005 mg/l	0.000266 mg/l
103-65-1	n-Propylbenzene	8260B	0.001 mg/l	0.000306 mg/l

100-42-5	Styrene	8260B	0.001 mg/l	0.000268 mg/l
630-20-6	1,1,1,2-Tetrachloroethane	8260B	0.001 mg/l	0.000467 mg/l
79-34-5	1,1,2,2-Tetrachloroethane	8260B	0.001 mg/l	0.000431 mg/l
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	8260B	0.001 mg/l	0.000335 mg/l
127-18-4	Tetrachloroethene	8260B	0.001 mg/l	0.000427 mg/l
108-88-3	Toluene	8260B	0.005 mg/l	0.00032 mg/l
87-61-6	1,2,3-Trichlorobenzene	8260B	0.001 mg/l	0.00037 mg/l
120-82-1	1,2,4-Trichlorobenzene	8260B	0.001 mg/l	0.00032 mg/l
71-55-6	1,1,1-Trichloroethane	8260B	0.001 mg/l	0.000375 mg/l
79-00-5	1,1,2-Trichloroethane	8260B	0.001 mg/l	0.0004 mg/l
79-01-6	Trichloroethene	8260B	0.001 mg/l	0.000263 mg/l
75-69-4	Trichlorofluoromethane	8260B	0.005 mg/l	0.000286 mg/l
96-18-4	1,2,3-Trichloropropane	8260B	0.001 mg/l	0.000464 mg/l
95-63-6	1,2,4-Trimethylbenzene	8260B	0.001 mg/l	0.000252 mg/l
526-73-8	1,2,3-Trimethylbenzene	8260B	0.001 mg/l	0.000252 mg/l
108-67-8	1,3,5-Trimethylbenzene	8260B	0.001 mg/l	0.000259 mg/l
75-01-4	Vinyl chloride	8260B	0.001 mg/l	0.000307 mg/l
1330-20-7	Xylenes, Total	8260B	0.003 mg/l	0.000728 mg/l

ATTACHMENT C

**ARIZONA DEPARTMENT OF HEALTH SERVICES ARIZONA DATA QUALIFIERS
GUIDANCE DOCUMENT**



[Office Home Page](#)

Office of Laboratory Services

Arizona Data Qualifiers Rev 2.0

**Arizona Data Qualifiers
Revision 2.0
11/26/2003**

Developed by the Sub-committee of the Arizona Environmental Laboratory Advisory Committee.

This is an updated list, with some new qualifiers added, some obsolete ones deleted and some modified, to the Rev 1.0 dated 05/31/2002. The new items are in bold.

The general guidelines for use and application of the following data qualifiers can be found as an attachment to this document (ATTACHMENT A).

Note: Using the Arizona Data Qualifiers does not automatically denote acceptability to the Regulatory Agency.

Microbiology:

A1 = Too numerous to count.

A2 = Sample incubation period exceeded method requirement.

A3 = Sample incubation period was shorter than method requirement.

A4 = Target organism detected in associated method blank.

A5 = Incubator/water bath temperature was outside method requirements.

A6 = Target organism not detected in associated positive control.

A7 = Micro sample received without adequate headspace.

Method/calibration blank:

B1 = Target analyte detected in method blank at or above the method reporting limit.

B2 = Non-target analyte detected in method blank and sample, producing interference.

B3 = Target analyte detected in calibration blank at or above the method reporting limit.

B4 = Target analyte detected in blank at/above method acceptance criteria.

B5 = Target analyte detected in method blank at or above the method reporting limit, but below trigger level or MCL.

B6 = Target analyte detected in calibration blank at or above the method reporting limit, but below trigger level or MCL.

B7 = Target analyte detected in method blank at or above method reporting limit. Concentration found in the sample was 10 times above the concentration found in the method blank.

Confirmation:

C1 = Confirmatory analysis not performed as required by the method.

~~C2 = Confirmatory analysis not performed. Confirmation of analyte presence established by site historical data.~~

C3 = Qualitative confirmation performed. ~~See case narrative.~~

C4 = Confirmatory analysis was past holding time.

C5 = Confirmatory analysis was past holding time. Original result not confirmed.

C6 = Sample RPD between the primary and confirmatory analysis exceeded 40%. Per EPA Method 8000B, the higher value was reported as there was no obvious chromatographic interference.

C7 = Sample RPD between the primary and confirmatory analysis exceeded 40%. Per EPA Method 8000B, the lower value was reported due to apparent chromatographic interference.

Dilution:

D1 = Sample required dilution due to matrix. ~~interference. See case narrative.~~

D2 = Sample required dilution due to high concentration of target analyte.

D3 = Sample dilution required due to insufficient sample.

D4 = Minimum reporting level (MRL) adjusted to reflect sample amount received and analyzed.

Estimated concentration:

E1 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not possible due to insufficient sample.

E2 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not performed due to sample matrix.

E3 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not performed due to holding time requirements.

E4 = Concentration estimated. Analyte was detected below laboratory minimum reporting level (MRL).

E5 = Concentration estimated. Analyte was detected below laboratory minimum reporting level (MRL), but not confirmed by alternate analysis.

E6 = Concentration estimated. Internal standard recoveries did not meet method acceptance criteria.

E7 = Concentration estimated. Internal standard recoveries did not meet laboratory acceptance criteria.

E8 = Analyte reported to MDL per project specification. Target analyte was not detected in the sample.

Hold time:

H1 = Sample analysis performed past holding time. ~~See case narrative.~~

H2 = Initial analysis within holding time. Reanalysis for the required dilution was past holding time.

H3 = Sample was received and analyzed past holding time.

H4 = Sample was extracted past required extraction holding time, but analyzed within analysis holding time. ~~See case narrative.~~

BOD:

K1 = The sample dilutions set-up for the BOD analysis did not meet the oxygen depletion criteria of at least 2 mg/L. Any reported result is an estimated value.

K2 = The sample dilutions set up for the BOD analysis did not meet the criteria of a residual dissolved oxygen of at least 1 mg/L. Any reported result is an estimated value.

~~K3 = The seed depletion was outside the method acceptance limits.~~

K4 = The seed depletion was outside the method ~~and laboratory~~ acceptance limits. The reported result is an estimated value.

K5 = The dilution water D.O. depletion was > 0.2 mg/L.

K6 = Glucose/glutamic acid BOD was below method acceptance criteria.

K7 = A discrepancy between the BOD and COD results has been verified by reanalysis of the sample for COD.

K8= Glucose/glutamic acid BOD was above method acceptance levels.

Laboratory fortified blank/blank spike:

L1 = The associated blank spike recovery was above laboratory acceptance limits. ~~See case narrative.~~

L2 = The associated blank spike recovery was below laboratory acceptance limits. ~~See case narrative.~~

L3 = The associated blank spike recovery was above method acceptance limits. ~~See case narrative.~~

L4 = The associated blank spike recovery was below method acceptance limits. ~~See case narrative.~~

~~Note: The L1, L2, L3 & L4 footnotes need to be added to all corresponding analytes for a sample.~~

Matrix spike:

M1 = Matrix spike recovery was high, the method control sample recovery was acceptable.

M2 = Matrix spike recovery was low, the method control sample recovery was acceptable.

M3 = The accuracy of the spike recovery value is reduced since the analyte concentration in the sample is disproportionate to spike level. The method control sample recovery was acceptable.

M4 = The analysis of the spiked sample required a dilution such that the spike concentration was diluted below the reporting limit. The method control sample recovery was acceptable.

M5 = Analyte concentration was determined by the method of standard addition (MSA).

M6= Matrix spike recovery was high. Data reported per ADEQ policy 0154.000.

M7= Matrix spike recovery was low. Data reported per ADEQ policy 0154.000.

General:

N1 = See case narrative.

N2 = See corrective action report.

N3 = The analysis meets all method requirements. See case narrative.

Sample quality:

Q1 = Sample integrity was not maintained. See case narrative.

Q2 = Sample received with head space.

Q3 = Sample received with improper chemical preservation.

Q4 = Sample received and analyzed without chemical preservation.

Q5 = Sample received with inadequate chemical preservation, but preserved by the laboratory.

Q6 = Sample was received above recommended temperature.

Q7 = Sample inadequately dechlorinated.

Q8 = Insufficient sample received to meet method QC requirements. **Batch** QC requirements ~~satisfies~~ ADEQ policies 0154 and 0155.

Q9 = Insufficient sample received to meet method QC requirements.

Q10 = Sample received in inappropriate sample container.

Q11 = Sample is heterogeneous. Sample homogeneity could not be readily achieved using routine laboratory practices.

Duplicates:

R1 = RPD exceeded the method control limit. See case narrative.

R2 = RPD exceeded the laboratory control limit. See case narrative.

~~R3 = Sample RPD between the primary and confirmatory analysis exceeded 40%. Per EPA Method 8000B, the higher value was reported.~~

R4 = MS/MSD RPD exceeded the method control limit. Recovery met acceptance criteria.

R5 = MS/MSD RPD exceeded the laboratory control limit. Recovery met acceptance criteria.

R6 = LFB/LFBD RPD exceeded the method control limit. Recovery met acceptance criteria.

R7 = LFB/LFBD RPD exceeded the laboratory control limit. Recovery met acceptance criteria.

R8 = Sample RPD exceeded the method control limit.

R9 = Sample RPD exceeded the laboratory control limit.

R10 = Sample RPD between the primary and confirmatory analysis exceeded 40%. Per EPA Method 8000B, the lower value was reported due to apparent chromatographic problems.

R11 = The RPD calculation for MS/MSD does not provide useful information due to the varying sample weights when Encore samplers/methanol field preserved samples are used.

Surrogate:

S1 = Surrogate recovery was above laboratory acceptance limits, but within method acceptance limits.

~~S2 = Surrogate recovery was above laboratory and method acceptance limits.~~

S3 = Surrogate recovery was above laboratory acceptance limits, but within method acceptance limits. No target analytes were detected in the sample.

S4 = Surrogate recovery was above laboratory and method acceptance limits. No target analytes were detected in the sample.

S5 = Surrogate recovery was below laboratory acceptance limits, but within method acceptance limits.

S6 = Surrogate recovery was below laboratory and method acceptance limits. Reextraction and/or reanalysis confirms low recovery caused by matrix effect.

S7 = Surrogate recovery was below laboratory and method acceptance limits. Unable to confirm matrix effect.

S8 = The analysis of the sample required a dilution such that the surrogate ~~concentration was diluted below the method acceptance criteria~~ **recovery calculation does not provide any useful information.** The method control sample recovery was acceptable.

~~S9 = The analysis of the sample required a dilution such that the surrogate concentration was diluted below the laboratory acceptance criteria. The method control sample recovery was acceptable.~~

S10 = Surrogate recovery was above laboratory and method acceptance limits. See Case narrative.

S11= Surrogate recovery was high. Data reported per ADEQ policy 0154.000.

S12= Surrogate recovery was low. Data reported per ADEQ policy 0154.000.

Method/analyte discrepancies:

T1 = Method ~~approved promulgated~~ by EPA, but not **yet licensed** by ADHS at this time.

T2 = Cited ADHS licensed method does not contain this analyte as part of method compound list.

T3 = Method not promulgated either by EPA or ADHS.

T4 = Tentatively identified compound. Concentration is estimated and based on the closest internal standard.

Calibration verification:

V1 = CCV recovery was above method acceptance limits. This target analyte was not detected in the sample.

V2 = CCV recovery was above method acceptance limits. This target analyte was detected in the sample. The sample could not be reanalyzed due to insufficient sample.

V3 = CCV recovery was above method acceptance limits. This target analyte was detected in the sample, but the sample was not reanalyzed. See case narrative.

V4 = CCV recovery was below method acceptance limits. The sample could not be reanalyzed due to insufficient sample.

V5 = CCV recovery after a group of samples was above acceptance limits. This target analyte was not detected in the sample. Acceptable per EPA Method 8000B.

V6= Data reported from one-point calibration criteria per ADEQ policy 0155.000.

V7= Calibration verification recovery was above the method control limit for this analyte, however the average % difference or % drift for all the analytes met method criteria.

V8= Calibration verification recovery was below the method control limit for this analyte, however the average % difference or % drift for all the analytes met method criteria.

Calibration:

W1= The % RSD for this compound was above ~~15% 20%~~. The average % RSD for all compounds in the calibration met the ~~15% 20%~~ criteria as specified in EPA method 8000B.

W2= The % RSD for this compound was above 15%. The average % RSD for all compounds in the calibration met the 15% criteria as specified in EPA method 8260B/8270C.

ATTACHMENT A

"Guidance on the Usage of Data Qualifiers"

These standardized data qualifiers are for use in qualifying analytical result for compliance samples in Arizona to represent events that occurred during analysis. The technical subcommittee has endeavored to develop qualifiers that are succinct and narrow in scope to eliminate broad or multiple interpretations when assessing the impact on data. It must also be noted that due to the specialized nature of the individual qualifiers, it is likely that more than one qualifier may be needed in order to accurately represent the data.

Note: Using the Arizona Data Qualifiers does not automatically denote acceptability to the Regulatory Agency.

Microbiology:

None.

Method/calibration blank:

Apply appropriate qualifier to affected analyte in the blank if target analyte is not detected at > RL in the samples. If analytes are detected, then all corresponding analytes for the associated samples should also be qualified.

Confirmation:

For methods that require qualitative confirmation. C3 applies to methods that require quantitative confirmation.

Dilution:

If all analytes are reported from the diluted sample, apply qualifier to the entire sample. Otherwise apply qualifier to each analyte that required dilution.

Estimated concentration:

Appropriate qualifier must be used for any analyte result reported outside the calibration range. Affects data reported outside the calibration range or down to the MDL. E8 is only required if additional clarification is necessary.

Hold time:

Qualify samples appropriately when method extraction and/ or analysis holding time have been exceeded.

BOD:

Qualifiers K4, K5, K6 & K8 indicate situations that may impact all results in an analytical run and should be used to qualify all affected samples as well as any affected quality control samples when reported. K3 was deleted because if the seed depletion was out, then the situation must be explained in the case narrative.

Laboratory fortified blank/blank spike:

Appropriate qualifier must be applied to the affected analytes in the Laboratory fortified blank/blank spike and to all corresponding analytes in the associated samples.

Matrix spike:

Appropriate qualifier must be applied to the affected analytes in the matrix spike and should also be added to all corresponding analytes in the associated spiked sample. If a batch spike recovery is outside of the acceptable range, it is permissible to only flag the sample that was spiked and not the other samples in the batch. As required in the Arizona Adopted Rules A.A.C. R9-14-617.F, clients must always be informed if the batch QC result is unacceptable whether one of their samples was spiked or not. The laboratory can choose how the unacceptable QC is reported to the client (e.g., cover letter or flag). The ADEQ policy 0154.000 can be accessed at <http://www.azdeq.gov/function/business/download/spike8.pdf>

General:

Use for events that cannot be described by the approved data qualifiers.

Sample quality:

Flag samples with appropriate qualifier when sample quality may be potentially impacted or when method requirements were not met.

The ADEQ policy 0154.000 can be accessed at

<http://www.azdeq.gov/function/business/download/spike8.pdf>

The ADEQ policy 0155.000 can be accessed at

http://www.azdeq.gov/function/business/download/one_pt3.pdf

Duplicates:

For use with sample, matrix spike, LFB and LCS duplicates. Qualify all affected analytes.

For MS/MSD or sample duplicates qualify only the original source sample.

Surrogate:

Qualify surrogates appropriately when they do not meet criteria. Surrogate failures in quality control samples will most likely require additional narration. S11 & S12 are used to qualify sample surrogates and only in cases where the Laboratory Fortified Blank/LCS has acceptable surrogate recoveries.

Method/analyte discrepancies:

For use with methods or analytes that are not currently approved under the Environmental Laboratory Licensure Rules.

Calibration Verification:

Appropriate qualifier must be applied to all affected analytes in any samples associated with the calibration verification.

The ADEQ policy 0155.000 can be accessed at

http://www.azdeq.gov/function/business/download/one_pt3.pdf

V7 and V8 are applicable to 8000 series methods only.

Calibration:

Any analytes reported utilizing a calibration per 'W1' and 'W2' data qualifiers must be qualified per method requirements.

[ADHS Web Privacy Policy](#), This page last modified on November 28, 2005.

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General comments, questions, or concerns: [ADHS Webmaster](#)

ATTACHMENT D

CHAIN OF CUSTODY FORMS AND LABORATORY WORK REQUEST FORMS



L-A-B S-C-I-E-N-C-E-S
12065 Lebanon Road
Mt. Juliet, TN 37122

Phone: (800) 767-5859
Phone: (615) 758-5858
Fax: (615) 758-5859

SPEEDIE & ASSOCIATES LABORATORY WORK REQUEST

PROJECT: _____ PROJECT NO: _____

PROJECT LOCATION: _____ LAB SAMPLE NO: _____

CLIENT: _____ SAMPLED BY: _____ SUBMITTED BY: _____
 Native Import

MATERIAL SOURCE: _____ DATE SAMPLED: _____ DATE SUBMITTED: _____
 ABC Sand Aggregate Asphalt Soil Other

MATERIAL DESCRIPTION: _____

SAMPLE LOCATION: _____

TESTS REQUIRED/AGENCY: _____ DUE DATE: _____

REQUESTED BY: _____ SAMPLE QUANTITY/CONTAINER: _____

APPENDIX C

HEALTH AND SAFETY PLAN



RCRA Facility Investigation (RFI) Work Plan
Former Romic Environmental Technologies Facility
Gila River Indian Community, Arizona

December 2011
212001

RFI WORK PLAN APPENDIX C

HEALTH AND SAFETY PLAN RCRA FACILITY INVESTIGATION AND CONTINUED GROUNDWATER SAMPLING

**FORMER ROMIC ENVIRONMENTAL TECHNOLOGIES FACILITY
LONE BUTTE INDUSTRIAL PARK
GILA RIVER INDIAN COMMUNITY, ARIZONA**

Prepared By

**Clear Creek Associates
6155 E. Indian School Rd., Suite 200
Scottsdale, Arizona 85251**

**December 2011
Project No. 212001**

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Plan-At-A-Glance

Safety Plan Summary Sheet

THIS SUMMARY SHEET IS PROVIDED AS A QUICK REFERENCE/OVERVIEW ONLY. THE REMAINDER OF THE SITE-SPECIFIC HEALTH AND SAFETY PLAN (HASP) IS INTEGRAL TO THE SAFE CONDUCT OF SITE OPERATIONS AND MUST BE APPLIED IN ITS ENTIRETY.

EMERGENCY INFORMATION:

Ambulance:	911
Fire:	911
Police	911
Hospital	(480) 728-3211 – Emergency Room Chandler Regional Hospital
Urgent Care	(480) 814-1560 – NON-THREATENING –NextCare Urgent Care

Clear Creek Assoc. Contacts:

Project Manager:	<u>Tom Suriano</u>	(480) 659-7131 (w) or (602) 762-0996 (m)
DSHO:	<u>Dave Giles</u>	(480) 659-7131 (w) or (602) 762-0676 (m)

Hospital: **Chandler Regional Hospital**

Telephone Number: (480) 728-3211 Emergency
Address: 475 S. Dobson Rd.
Chandler, AZ 85224

Hospital Directions:

Leave site heading EAST (left) on Allison Road, to Kyrene Rd. (~0.67 mi.). Take Kyrene Road NORTH (left) to Chandler Blvd. (~ 1.2 mi.). Take Chandler Blvd. EAST (right) to Dobson Road (~4 mi.). Take Dobson Road SOUTH (right) to Chandler Regional Hospital ER – for a LIFE THREATENING injury OR NextCare Urgent Care Center – for NON-LIFE THREATENING Injury.

CONSTITUENTS OF CONCERN:

Trichloroethene 36 µg/L maximum in groundwater (August 2011)
Tetrachloroethene 65 µg/L maximum in groundwater (August 2011)
1,1-DCE 6.6 µg/L maximum in groundwater (August 2011)



1.0 INTRODUCTION

It is Clear Creek Associates' (Clear Creek) intent to provide a safe work environment for employees and subcontractors. This Health and Safety Plan (HASP) has been developed to fulfill the following objectives:

1. Instruct employees and subcontractors on procedures to minimize the potential for injury or exposure to a hazardous condition.
2. Train employees and subcontractors on the proper action to be taken if a hazardous condition cannot be avoided by engineering controls.
3. Provide guidelines for emergency response for known hazards and hazardous situations.
4. Specify actions required to comply with applicable (1) Occupational Safety and Health Administration (OSHA) regulations and (2) state and local regulations or other requirements.
5. Present subcontractors activity-specific safety and health practices and procedures for site hazards and activities unique to the subcontractors' operations, (e.g., drilling).

Note that this HASP is designed specifically for the drilling and soil sampling in the vicinity of a hazardous waste transfer facility. This HASP is not intended for projects involving hazardous materials. If hazardous materials are encountered during field activities, work must cease until the situation is re-evaluated and a hazardous materials HASP is written for the site.

1.1 Purpose of the Site Safety and Health Plan

This HASP is intended as a guideline that allows the Site Safety and Health Officer (SSHO) to respond to changing conditions and make professional judgments regarding the interpretation of monitoring data and related control measures. This HASP also delineates Safety and Health responsibilities and assigns those responsibilities to project and office personnel. This document is to be read and understood by site personnel. The specific requirements of this HASP apply to Clear Creek employees and subcontractors involved in implementing the described scope of work. It is not applicable to other contractors and/or site tasks unless authorized for such use by a designated Clear Creek representative. Site personnel (employees and subcontractors) must be permitted access to this HASP upon request. Site personnel are required to sign this HASP as an acknowledgment of agreement, acceptance, and understanding of the contents.

1.2 Scope of Work

Clear Creek will subcontract an Arizona licensed driller to drill a series of boreholes, collect subsurface soil and groundwater samples from each borehole and potentially install groundwater monitoring well(s) in select borings. The investigation is being performed to further evaluate the groundwater conditions in the vicinity of the former Romic Environmental Technologies Corporation facility located at 6760 West Allison Road in the Lone Butte Industrial Park on the Gila River Indian Community (GRIC). The borings will be drilled using either percussion hammer or rotosonic drilling techniques. Based on the measured depths to water at the site, it is expected that groundwater will be encountered at approximately 70-to-75 feet below ground surface (bgs) and the expected total depth of the borings will be 140 feet bgs.

1.3 Implementation and Modification of the Site Safety and Health Plan

Before any activities begin on or around the site, a safety and health tailgate meeting will be held with site personnel to discuss safety procedures and to familiarize personnel with the potential hazards of the site. Changes in this HASP will be discussed with the Designated Safety and Health Officer (DSHO) before being applied at the site. Site personnel will be informed of changes during the weekly tailgate meetings or when site conditions or risks change.



The SSHO will perform daily inspections of the site dependent on field activities. If any operation, practice, or equipment does not pass inspection, the SSHO will document the item in the logbook and initiate corrective actions. The SSHO will notify the DSHO of inspection findings as appropriate. Operations will cease or the faulty equipment will be removed, as appropriate. Unacceptable practices and/or faulty equipment will be remedied immediately, and the HASP will be modified to correct any deficiencies.



1.4 Approval of the Site Safety and Health Plan

Plan Prepared By:	Geno Mammini Name	Project Hydrogeologist Title
	Signature	Date
Plan Approved By:	Tom Suriano Name	Principal Hydrogeologist Title
	Signature	Date
DSHO	David Giles Name	Senior Project Manager Title
	Signature	Date
Plan Revised By:	Name	Title
	Signature	Date
	Name	Title
	Signature	Date

2.0 SITE AND PROJECT INFORMATION

This section provides a general description of the site and a discussion of chemicals previously detected in onsite media and chemicals suspected to be present onsite.

2.1 General Site Description

The site is an inactive former transportation, storage and disposal (TSD) facility managing new and reclaimed solvents and industrial chemicals.

2.1.1 Site Status

Active/Open Inactive/Open Inactive/Closed Unknown

2.1.2 Site History

The former Romic Facility is located 6760 W. Allison Road, roughly 0.4 mi east of the intersection of Allison Road and 56th Street/Maricopa Road, within the Lone Butte Industrial Complex. The site is under lease from the Gila River Indian Community (GRIC).

2.1.3 Surroundings

The site is located within the Lone Butte Industrial Complex, in the Gila River Indian Community, south of Chandler, Arizona

2.1.4 Climate

Average Wind Speed and Direction: 5-15 knots from the east in the mornings and the west afternoon.

Humidity: Arid Semiarid Humid Tropical

Expected High Temperature (°F) 115°

Expected Low Temperature (°F) 70°

2.1.5 Locations of Resources Available to Onsite Personnel

Drinking water supply: On site

Telephone: With Staff

Radio: N/A

2.2 Chemicals in Onsite Media

Chemicals identified as present onsite, the media in which they were detected, and the maximum detected concentrations are listed in Table 1. Hazardous property information for the chemicals listed in Table 1 is provided in Attachment A.

2.3 Site Work Zones

Site zones may be established as necessary to prevent or minimize exposure (of unauthorized personnel) to hazards by establishing boundaries to reduce migration of contaminants into clean areas. For this site, it is expected that work will be conducted under nonhazardous conditions. However, if a hazardous condition is identified, a three-zone approach will be implemented for field activities as appropriate. The zones will be identified during safety briefings and will be clearly marked by traffic cones, fencing, barricades, signs, or other means. These three zones shall be designated as the Support Zone, the Contamination Reduction Zone, and the Exclusion Zone. Site entrance and exit shall be through controlled access points established for each work location.

2.3.1 Support Zone (SZ)

The Support Zone is the clean area in which the possibility of encountering hazardous materials or conditions is minimal. Therefore, personal protective and respiratory equipment are not necessary. Inside the Support Zone, the following will be available: an effective means of communication, first-aid supplies, fire extinguisher, drinking water, and other appropriate support equipment. The Support Zone shall also serve as the main point of contact for the visitor check-in and initiation of emergency services when necessary.

2.3.2 Contamination Reduction Zone (CRZ)

The Contamination Reduction Zone is the area where equipment and personnel are decontaminated before leaving the Exclusion Zone. Personnel will remove and/or decontaminate personal protective equipment (PPE) and place it in appropriate containers. Site vehicles and equipment will also be decontaminated in the Contamination Reduction Zone. The Contamination Reduction Zone will consist of a means of washing protective equipment, site vehicles, and equipment; containers for liquids, solids, and PPE; first-aid supplies; an eyewash/emergency shower; and a fire extinguisher.

2.3.4 Exclusion Zone (EZ)

The Exclusion Zone includes the work activities at the site (e.g., drilling, sampling, etc.). Only authorized, trained, and qualified personnel with the appropriate personal and respiratory equipment shall be admitted. Personnel entering the Exclusion Zone must use the buddy system.

Work activities within the Exclusion Zone pose the greatest possibility of exposure to personnel and equipment. The Site Supervisor shall be responsible for controlling the access points to the Exclusion Zone and Contamination Reduction Zone and keeping bystanders and unauthorized personnel to a minimum. The Exclusion Zone will be clearly marked with flagging, fencing, barricade tape, traffic cones, or other indicators to limit access.

3.0 PROJECT ORGANIZATION

This section presents discussions of safety and health responsibilities of key personnel and CCA personnel requirements.

3.1 Organization and Safety Responsibilities

To meet its safety and health objectives, CCA has developed a line of reporting and tasked individuals with safety and health responsibilities. This information is presented below.

OFFICE MANAGER: **Lori Bartlett**

To receive, consider, and initiate action upon recommendations from project personnel and/or DSHO. Overall responsibility for the implementation and effectiveness of the Clear Creek Safety and Health Program.

PROJECT MANAGER: **Tom Suriano**

Acquaint field personnel with potential hazards and procedures to minimize the negative impact of those hazards. Make available proper PPE, adequate time and budget, and trained personnel to perform site work in a safe manner. Arrange for preparation of an HASP. Investigate and report to the DSHO each work-related illness or injury, near-misses, accidents, and damage to physical property.

DESIGNATED SAFETY AND HEALTH OFFICER: **David Giles**

Write or review and approve the HASP. Implement safety and health procedures that are stated in the HASP. Conduct periodic audits to confirm that the HASP is being followed.

SITE SUPERVISOR: **Geno Mammini**

Ensure that site personnel have read and signed the master copy of this document (Attachment B). Coordinate with the SSHO regarding accident investigations, as necessary. An accident investigation form is included as Attachment C.

SITE SAFETY AND HEALTH OFFICER: **Geno Mammini**

Ensure that the guidelines, rules, and procedures in this document are followed for site work. Check that site personnel meet OSHA requirements regarding training, medical examinations, and fit testing. Be familiar with local emergency services. Conduct a tailgate safety and health meeting before work startup and weekly thereafter. Additional meetings may be required for specific job tasks or site activities. Maintain and inspect PPE, monitor onsite hazards, and monitor the physical condition of site personnel. Perform daily inspections of work site activities. Maintain safety and health files, which will include training and medical certifications, tailgate meeting notes and rosters, inspection reports, or other safety and health documentation, as applicable. Shut down operations that pose a potential threat to site personnel.

EMPLOYEES

Obey safety and health work practices issued by law and by Clear Creek. Wear PPE as directed by this HASP. Use safety equipment as directed by this HASP.

VISITORS

Follow the direction of the Site Supervisor or the SSHO. Read, understand, and sign the HASP. Do not enter the work zones unless the appropriate OSHA-required training and medical monitoring has been obtained. Use PPE, as appropriate.

SUBCONTRACTORS

Follow the guidelines, rules, and procedures in this document. Attend tailgate meetings and sign the HASP following each meeting. Report recognized unsafe conditions and actions to the SSHO and/or the Project Manager. Provide Material Safety Data Sheets (MSDSs) for subcontractor-provided materials at the job site. Provide their

own safety and health procedures addressing hazard recognition, evaluation and control practices/procedures for general and specific site hazards and activities unique to their operations.



4.0 CLEAR CREEK ASSOCIATES' SAFETY AND HEALTH PROGRAMS

Required Clear Creek safety and health programs, including training and medical monitoring, respiratory protection, and hazard communication are presented in this section.

4.1 Required Personnel Training

Specific training requirements for personnel, including subcontractors conducting field activities, are divided into the following training categories:

- Regular Site Personnel Exposed to Hazardous Substances
- Regular Site Personnel Potentially Exposed to Hazardous Substances below Permissible Exposure Limits
- Occasional Site Personnel Potentially Exposed to Hazardous Substances below Permissible Exposure Limits
- 40 hour OSHA Safety and Health Training
- Management and Supervisory Training
- Refresher Training
- Specific Hazard or Equipment Training

Regular and occasional site personnel will be present for the tasks identified in the scope of work. The tasks identified in the scope of work for this project require 40 hour OSHA Safety and Health training, 8 hour refresher training, and, for the Site Safety and Health Officer and the site manager, Management and Supervisor Training. Training will also be given to personnel working around specific hazards or equipment. Staff working on this project will have training appropriate to their job description. Site specific training will be conducted for specific site hazards, equipment, and station safety requirements.

These categories, as well as documentation, exempt personnel, tailgate meetings, and audits, are discussed in the following sections.

4.1.1 Regular Site Personnel Exposed to Hazardous Substances

Site personnel whose job responsibilities cause them to be exposed to or to have the potential to be exposed to hazardous substances or health hazards are required to comply with 29 Code of Federal Regulations (CFR) Section 1910.120(e)(3)(I) or applicable state regulations. This regulation requires site personnel exposed to hazardous substances to complete 40 hours of offsite instruction and three days of field experience supervised by a trained supervisor.

4.1.2 Regular Site Personnel Potentially Exposed to Hazardous Substances Below Permissible Exposure Limits

Regular site personnel whose job responsibilities cause them to be potentially exposed to hazardous substances below permissible exposure limits (PELs) or health hazards are required to comply with 29 CFR Section 1910.120(e)(3)(iii) or applicable state regulations. This regulation requires that these personnel receive a minimum of 40 hours of offsite instruction and one day of field experience supervised by a trained supervisor. The project SSHO or designated representative must ensure that these personnel will not be exposed above PELs. This decision will be made on the basis of review of previous monitoring in these work areas and possibly historical site background information.

4.1.3 Occasional Site Personnel Potentially Exposed to Hazardous Substances Below

Permissible Exposure Limits

Occasional site personnel who visit the site for a specific limited task and whose exposure is designated by the SSHO to be under PELs are required to comply with 29 CFR Section 1910.120(e)(3)(ii) or applicable state regulations. This regulation requires that these personnel receive a minimum of 40 hours of offsite instruction and one day of field experience supervised by a trained supervisor.

In accordance with 29 CFR Section 1910.120(e)(3)(iv) or applicable state regulations, regular (as defined in Section 4.1.2 above) and occasional site personnel having completed an initial 24-hour classroom instruction must complete an additional 16 hours of offsite instruction and two days of field experience supervised by a trained supervisor before they are qualified to engage in activities that may expose them to hazardous substances above PELs.

4.1.4 Management and Supervisory Training

In accordance with 29 CFR Section 1910.120(e)(4) or applicable state regulations, individuals who manage or supervise personnel engaged in hazardous waste operations at the site must receive 40 hours of offsite instruction and three days of field experience supervised by a trained supervisor. In addition, management and supervisory personnel shall receive an additional 8 hours of specialized training that addresses the safety and health program, training requirements, personal protective and respiratory equipment program, health hazard monitoring procedures, accident investigation, and emergency response procedures.

4.1.5 Refresher Training

Annual refresher training in accordance with 29 CFR Section 1910.120(e)(8) or applicable state regulations shall be completed at least annually following the completion of the individual's 40-hour or 24-hour training course. Personnel will be required to attend the annual refresher training to maintain their qualifications for hazardous waste operation.

4.1.6 Documentation

Training must be properly documented and filed onsite for reference by the SSHO or designated representative. Personnel required to meet the training requirements must present evidence of this training at the site. The Site Supervisor is responsible for checking before each activity to verify complete and current documentation. A copy of the documentation will be kept readily available or onsite, as applicable.

4.1.7 Exempt Personnel

Exempt personnel requesting access to the site could include personnel making deliveries or performing repairs to utilities, public or government officials, untrained visitors, or local residents. Individuals from these groups will not be required to comply with the training requirements as previously stated or the medical monitoring as discussed in Section 4.2. However, access will be limited to designated work, delivery, or observation areas onsite to minimize any potential exposure. Observation areas onsite will be upwind from site operations, as decided on the basis of predominant wind directions, so as not to receive dust or chemical contaminants. Access to observation areas may be restricted by weather conditions or site activities. Approvals for exempting personnel and decisions on access limitation for other personnel will be handled on a case-by-case basis by the Site Supervisor in consultation with the SSHO and CCA Project Manager.

4.1.8 Site-Specific Training

Site-specific training is not required for this project.

4.1.9 Tailgate Safety Meetings

A tailgate safety meeting shall be conducted each day before starting work or (1) whenever risks or hazards change, (2) whenever new personnel arrive, and (3) when site operations warrant indoctrination and training. Safety meetings shall be conducted by the Site Supervisor or another qualified individual. Where procedural deficiencies are identified, additional safety meetings will be conducted to address the situation. The following should be addressed during the meetings:

- Review of planned activities
- Hazards suspected
- PPE required
- Communications
- Site personnel responsibilities
- Decontamination procedures
- Emergency procedures

These tailgate safety meetings will be documented on the appropriate form in Attachment B.

4.1.10 Safety Inspections and Audits

The Site Supervisor will inspect the site daily to identify potential hazardous conditions or work areas. The DSHO will visit the site periodically to ensure that work operations are being conducted in compliance with the protocols and procedures outlined in this HASP.

4.2 Medical Monitoring

Clear Creek field employees working at hazardous sites more than 30 days per year will receive a baseline and annual comprehensive medical evaluation to qualify for hazardous waste site assignments and to monitor work-related illness or contamination. Other employees who are exposed to hazardous substances or waste or who participate in physically challenging work will receive a baseline and periodic exams (less frequently than annually).

Any employee who suffers an illness or injury that imposes a medical restriction on his or her job duties must have a physician's release statement indicating that he or she is fit for duty before the SSHO will permit that employee to return to full duty.

Medical monitoring will be required for personnel at the site, including visitors, subcontractors, client representative, U.S. Environmental Protection Agency (EPA) officials, and others visiting the works sites who may be exposed to contaminants exceeding accepted PELs. Clear Creek is responsible for providing medical monitoring to Clear Creek personnel only. Clear Creek is not responsible for providing medical monitoring for other parties visiting the site. However, Clear Creek may review visitor certifications to assess whether the monitoring is up to date.

4.3 Respiratory Protection Policy

Clear Creek's respiratory protection program is managed by the DSHOs of the Phoenix and Tucson offices. The purposes of the program are as follows:

- Provide adequate respiratory protection to site personnel where there is a potential for exposure to toxic or nuisance substances in excess of allowable concentrations.
- Require the provision of adequate respiratory equipment to CCA employees who may request such equipment.
- Determine that employees assigned to site work requiring respiratory protection are physically able to wear respiratory protection equipment based on the results of medical monitoring.
- Protect the employee's health during normal job duties.

Objectives of the respiratory protection program are as follows:

- Address the site hazards, the need for respiratory protection, and the selection of the appropriate National Institute for Occupational Safety and Health (NIOSH) or Mine Safety and Health Administration (MSHA)-approved equipment during preparation of this HASP.
- Use engineering controls at the work site to minimize the potential for exposure. If engineering controls are not feasible, respiratory equipment must be used.
- Fit test employees required to wear respirators using isoamyl acetate and/or irritant smoke or a quantitative fit test. Testing shall be conducted annually for normal fieldwork or every six months for asbestos work. Records are maintained by the DSHOs in each office regarding whether the employee passed the fit test and what type and size respirator he or she is assigned.
- Inspect, maintain, sanitize, and appropriately store respirators, as determined by the DSHOs.
- Site visitors, subcontractors, or others who may request entry into the Exclusion Zone must show proof of current (annual) respirator fit testing. Copies of this documentation will be kept readily available onsite, as applicable.

4.4 Hazard Communication

The DSHO is responsible for administering the program. The hazard communication program governs "hazardous substances" and excludes "hazardous waste."

4.4.1 Container Labeling

Clear Creek requires that containers and secondary containers of hazardous substances both in the office and at the job site be labeled as to the contents, appropriate hazard warning, and the name and address of the manufacturer. The DSHO and the SSHO are responsible for label verification for this project.

4.4.2 Material Safety Data Sheets

MSDSs are obtained from the manufacturer when hazardous substances are purchased by Clear Creek or a Clear Creek subcontractor to conduct field activities. If the manufacturer does not include the MSDS when the item is shipped, the manufacturer will be contacted by telephone for a facsimile transmittal of the MSDS. Additionally, the item will not be used until an MSDS is received. The MSDSs are kept in the each HASP, as applicable. In addition to each employee's initial hazard communication training, significant safety and health information for each hazardous substance is made available to affected employees. Site personnel may request any or all MSDSs for review at any time. A listing of the hazardous substances in use is filed with the HASP.

5.0 PROJECT HAZARD IDENTIFICATION AND MITIGATION

This section discusses general safety and health work practices and hazard identification and mitigation. PPE and monitoring instruments that will be used onsite are also discussed. See Tables 2 and 3 for information regarding Hazard Analysis and Risk Assessment.

5.1 General Safety and Health Work Practices

Clear Creek's general safety policy advocates exercising every reasonable precaution when performing the work to prevent property damage and to protect the safety and health of employees, the public, and the environment.

Employees have certain responsibilities for their own safety, as follows:

- Report to work rested, and physically and mentally fit to perform the job assignment.
- Working while under the influence of intoxicants, narcotics, or controlled substances is prohibited.
- Wear suitable clothing for the weather and the work.
- Wear PPE and follow established procedures for a particular job. Do not wear jewelry or loose-fitting clothing when operating or near equipment.
- Call the supervisor's attention to any behavior or condition that may cause injury or illness to others or damage to property.
- Read warning labels on containers and equipment. Follow specified precautions.
- Discontinue any operation that could lead to injury, illness, or property damage.
- Keep horseplay and other disruptive behavior away from the job.
- Promptly report to the Site Supervisor, SSHO, or DSHO any occupational injury, illness, or exposure to toxic material. If injured, get first aid. Small injuries can become serious if neglected.
- Promptly inform the Site Supervisor, SSHO, or DSHO whenever new substances, processes, procedures, or equipment that could present new safety and health hazards are brought into work areas or onto projects.
- Do not eat, smoke and/or chew tobacco, or chew gum in the work area.
- Do not allow visitors without adequate safety training or personal protective equipment into the work area.
- Work upwind of field activities when it is possible to do so.
- Perform work in a manner that will minimize dust from becoming airborne (i.e., use water spray or wet technique when feasible).
- Do not work alone inside the CRZ or Exclusion Zone work area.
- Enter the CRZ and EZ work areas only while in proper PPE and with a "buddy." The buddy system will also be in effect at any work zone where respirators are being worn.
- Avoid contact with objects or water unless the contact is necessary to the field operation.
- Be alert to any abnormal behavior of other personnel that may indicate distress, disorientation, or other ill effects.
- Verify that vehicles have an ABC-rated fire extinguisher, a first-aid kit, and 32 ounces of eyewash fluid.
- Monitor weather conditions, particularly wind direction, because they could affect potential exposure.
- Operate a vehicle only if you are a licensed driver. Seatbelts must be worn when operating a company vehicle or when driving a private vehicle on company business.
- Drive company vehicles safely and professionally and care for them as you would other company property. Drive only vehicles that are safe and within maintenance specifications. Obey traffic regulations.
- Do not exceed speed limits for conditions.
- Practice defensive driving.
- Park in legal spaces; do not obstruct traffic.
- Lock vehicle when unattended.
- Contact the DSHO if contact with human blood occurs during the administration of first aid.



These general safety responsibilities also apply to subcontractors and visitors.

5.2 Project Hazard Analysis

This section provides information regarding potential hazards that might be encountered during field activities and the risk(s) associated with each hazard. Projects are identified by activity numbers as follows:

Activity Number	Job Project
1	Drilling
2	Well Installation
3	Groundwater Monitoring – Water levels
4	Groundwater Monitoring - Sampling
NA	Not applicable to this Project.

The hazard analysis evaluates the possible type of hazards at the site by activity. The analysis is presented in Table 3.

5.3 Hazard Mitigation

Procedures that will be used to minimize hazards identified onsite are listed in this section.

Hazards	Procedures to Mitigate Hazards
Physical	
	<u>1,2,3,4</u> Keep ground clutter in the work area to a minimum to minimize the potential for tripping.
	<u>1,2,3,4</u> Use caution when the ground surface is slick or uneven.
	<u>1,2,3,4</u> Clean up liquid spills or use sorbent material (e.g., dry soil) to minimize the potential for loss of footing.
	<u>1,2,3,4</u> Exercise caution when using hand tools.
	<u>1,2,3,4</u> Use proper lifting and reaching techniques.
	<u>1,2,3,4</u> Use hand carts or ask for assistance when lifting or moving heavy loads.
	<u>1,2,3,4</u> Wear safety vests and be aware of motorized traffic when working in roadways or on road shoulders.
Mechanical	
	<u>NA</u> Do not stand near backhoe buckets and earthmoving equipment.
	<u>1,2</u> Establish communication hand signals with heavy equipment operators.
	<u>1,2,3,4</u> Verify daily that all equipment and associated tools are in good condition.
	<u>1,2</u> Identify the drilling rig kill switches to personnel.
	<u>1,2</u> Do not stand or walk under elevated loads or ladders without appropriate guarding.

Hazards	Procedures to Mitigate Hazards
	<u>1,2,4</u> Do not repair equipment while it is in operation.
	<u>1,2,3,4</u> Immediately remove defective equipment from the work site for subsequent repair.
	<u>1,2,3,4</u> Wear a seat belt if you are a heavy equipment operator or a motor vehicle occupant.
	<u>1,2</u> Do not ride on the forks of lift truck, backhoe, or front-end loader buckets, or on a load, rigging, hook, or ball.
	<u>1,2,3,4</u> Inspect motor vehicles and heavy equipment before operating.
	<u>1,2,4</u> Do not wear loose clothing near operating equipment. Tie back long hair.
	<u>1,2,3,4</u> Consult the DSHO if other mechanical hazards exist.
Trenching/Excavating	
	<u>N/A</u> Do not stand near unguarded excavations or trenches.
	<u>N/A</u> Do not enter excavations or trenches greater than 5 feet deep that are not properly guarded, shored, or sloped, as specified in 29 CFR 1926, Subpart P, or applicable state regulations.
	<u>N/A</u> Use a protection system if personnel will be entering a trench/excavation greater than 4 feet deep. Provide a description of the system.
	<u>1</u> Locate and mark buried utilities.
	<u>1</u> Locate spoils, other materials, and equipment at least 2 feet from the side of the excavation.
	<u>N/A</u> Ensure that daily inspections of the excavations are performed by a competent person.
	<u>1</u> Avoid using an existing wall or structure until it has been determined to be safe.
	<u>1</u> Barricade, cover, or backfill wells, pits, and shafts when the operation is complete.
	<u>N/A</u> Use diversion ditches, dikes, or other means to prevent water from entering the excavation.
	<u>N/A</u> Ensure that work in trenches and excavations is supervised by a qualified person (one knowledgeable in soil conditions and trenching and excavation safety).
	<u>N/A</u> Ensure that trenches 4 feet deep or greater have an adequate means of exit, such as ladders or steps, with no more than 25 feet of lateral travel.
Electrical	

Hazards	Procedures to Mitigate Hazards
	<p><u>1</u> Locate and mark buried utilities before drilling or excavating.</p> <p><u>1</u> Utilities located by _____ on _____. (To be completed by the Site Supervisor when location is complete.)</p>
	<p><u>1,2,3,4</u> Obtain permits, licenses, or right of entry required by local or state authorities.</p>
	<p><u>1,2</u> Maintain at least a 20-foot clearance (or minimum recommended by the local utility company) from overhead power lines. Contact the client for additional requirements.</p>
	<p><u>1,2</u> Contact the utility company for information regarding minimum clearance from high-voltage power lines.</p>
	<p><u>1,2</u> If unavoidably close to buried or overhead power lines, have power turned off, with circuit breaker locked and tagged or have the local utility company mask the wires.</p>
	<p><u>1,2,4</u> Properly ground electrical equipment. Use only three-wire grounded receptacles and extension cords.</p>
	<p><u>1,2,4</u> Do not stand in water when operating electrical equipment.</p>
	<p><u>1,2,4</u> If equipment must be connected by splicing wires, be sure connections are properly taped and that the splice is equal to the cord's quality.</p>
	<p><u>1,2,3,4</u> Be familiar with specific operating instructions for each piece of equipment.</p>
	<p><u>1,2,3,4</u> Consider all wires live until locked and tagged out.</p>
	<p><u>1,2,4</u> Use ground fault circuit interrupters (GFCIs) if operating outdoor electrical equipment.</p>
Chemical	<p><u>1,2,3,4</u> Use PPE as indicated in Section 5.4 or as directed by the SSHO.</p>
	<p><u>1,2,3,4</u> Conduct direct-reading air monitoring to evaluate respiratory and explosion hazards (list instrument, action level, monitoring location, and action to be taken in Section 5.5).</p>
	<p><u>1</u> Locate underground pipelines before drilling or excavating.</p>
	<p><u>1,2,3,4</u> Do not use spark-ignition equipment when in a flammable or combustible environment.</p>
	<p><u>1,2,3,4</u> Do not smoke, except in designated areas.</p>
	<p><u>1,2,3,4</u> Use fans to disperse airborne contaminants at the work site as necessary.</p>
	<p><u>1,2,3,4</u> No sparking or open flame equipment will be permitted inside the work area if there is a potential of reaching the Lower Explosive Limit (LEL) of contaminants</p>

Hazards	Procedures to Mitigate Hazards
	present at the site.
<u>1,2,3,4</u>	Respirators with high-efficiency particulate/air (HEPA) filters should be worn if there is a potential for contaminated dust at the site.
<u>1,2,3,4</u>	Consult the DSHO or SSHO for personal air monitoring.
Temperature Heat stress	
<u>1,2,3,4</u>	When the temperature exceeds 70 degrees Fahrenheit (°F), take frequent breaks in shaded areas. Use the buddy system.
<u>1,2,3,4</u>	Unzip or remove coveralls during breaks.
<u>1,2,3,4</u>	Have cool water or electrolyte replenishment solution available. Drink small amounts of cool water or electrolyte replenishment solution frequently to avoid dehydration.
<u>1,2,3,4</u>	Count the pulse rate for 30 seconds as early as possible in the rest period. If the pulse rate exceeds 110 beats per minute at the beginning of the rest period, shorten the work cycle by one-third.
<u>1,2,3,4</u>	If the internal temperature exceeds 99.6°F, shorten the next work cycle by one third. Personnel wearing Modified D or Level C PPE are not permitted to continue work if their temperature meets or exceeds 100.6°F.
Cold stress	
<u>1,2,3,4</u>	Include salt in your food intake. Salt tablets are not recommended.
<u>NA</u>	Wear multilayer cold-weather clothing. The outer layer should be wind-resistant fabric.
<u>NA</u>	Limit total work time in 0° to -30°F to four hours. Alternate one hour in and one hour out of the low-temperature area. Below -30°F, consult the DSHO.
<u>NA</u>	Drink warm fluid and take rest breaks in a warm shelter. Use the buddy system. Avoid heavy sweating.
Acoustical	
<u>1,2,4</u>	Post appropriate signs in areas of hazardous noise.
<u>1,2,4</u>	Use earplugs or earmuffs when noise prevents conversation in a normal voice at a distance of 3 feet. (This is a "rule of thumb" that indicates noise levels are exceeding 85 decibels.) Double protection (e.g., earplugs and muffs) must be used for noises exceeding 104 decibels.
<u>1,2,4</u>	Contact the DSHO for a noise survey, as needed.
Confined Spaces	
<u>NA</u>	Confined spaces include trenches, pits, sumps, elevator shafts, tunnels, or any other area that is not designated for human occupancy, where circulation of fresh air is restricted, or where the ability to readily escape from the area is restricted.

Hazards	Procedures to Mitigate Hazards
	<p>Consult the DSHO and CCA Safety and Health Policy and Procedures Manual before entering a confined space.</p>
<u>NA</u>	<p>The SSHO will consult with the DSHO to assess whether a confined space is a permitted or nonpermitted confined space. If it is a permitted confined space, a permit must be obtained from the DSHO before entering.</p>
<u>NA</u>	<p>Monitor oxygen (O₂), LEL, and organic vapors before entering a confined space. If the following values are exceeded, DO NOT ENTER:</p> <p>O₂ less than 19.5 percent or more than 23.5 percent</p> <p>Total hydrocarbons greater than 5 parts per million (ppm) above background, if all air contaminants are not identified</p> <p>Concentrations of specific contaminants exceeding action levels in Section 5.5.1 if all air contaminants are identified</p> <p>Flammable gases more than 10 percent of the LEL</p>
<u>NA</u>	<p>Monitor O₂, LEL, and organic vapors continuously while inside a confined space. If Threshold Limit Values (TLVs) cited in Appendix A are exceeded, EVACUATE IMMEDIATELY. Record instrument readings.</p>
<u>NA</u>	<p>Ensure that at least one person capable of pulling personnel from the confined space is on standby outside the confined space. The observer must be trained in cardiopulmonary resuscitation (CPR) and first aid.</p>
<u>NA</u>	<p>Use portable fans or blowers to introduce fresh air to confined spaces whenever respirator use is required.</p>
<u>NA</u>	<p>Do not perform work that involves the use of flame, arc, spark, or other source of ignition in a confined space that contains a hazardous atmosphere.</p>
Natural	<p><u>1,2,3,4</u> Wear long sleeves and/or sun block on sunny days.</p> <p><u>1,2,3,4</u> Cease field activities during severe storms. Seek shelter until the storm has passed.</p> <p><u>1,2,3,4</u> If a tornado occurs: Seek shelter in the lowest level or interior wall of a substantial building. Do not seek shelter in a trailer or vehicle. Lie flat in the nearest ditch.</p> <p>If an earthquake occurs while working outside: Stay away from buildings, trees, and power lines.</p> <p><u>1,2,3,4</u> If operating a motor vehicle or heavy equipment, stop immediately but stay in the vehicle or piece of equipment until the tremors have stopped.</p>

Hazards	Procedures to Mitigate Hazards
	<u>1,2,3,4</u> See Section 7.0 for further information.
Biological	<u>1,2,3,4</u> Learn to recognize toxic plants, such as poison oak, poison ivy, and poison sumac. <u>1,2,3,4</u> Wear long-sleeved shirts, sturdy trousers, and boots when working near toxic plants to minimize the potential of skin contact. <u>1,2,3,4</u> If exposed to toxic plants, shower as soon as possible with a strong soap. <u>1,2,3,4</u> Do not touch plants that have hairy leaves, milky sap, or thorny leaves, or fruit or seed pods. <u>1,2,3,4</u> Do not approach or agitate animals, especially ones behaving strangely or foaming at the mouth. <u>1,2,3,4</u> Avoid breathing dust in dry desert or central valley areas (may cause valley fever). <u>1,2,3,4</u> Use insect repellent to avoid contact with ticks, mosquitoes, and other insects (disease carriers or poisonous), as necessary. Use a solid repellent to minimize potential contamination of field samples. <u>1,2,3,4</u> If possible, avoid contact with poisonous snakes or other reptiles by quietly walking away. If bitten, seek medical assistance immediately. <u>1,2,3,4</u> Avoid contact with rodents because they frequently are hosts to fleas, which can carry typhus and the plague. Rodent urine may also contain spirochetes harmful to human health. <u>1,2,3,4</u> Avoid encounters with stinging insects.
Fire/Explosion	<u>1,2,3,4</u> Use a fire extinguisher only to escape or to fight very small fires. Do not attempt to fight large fires. <u>1,2,3,4</u> Field vehicles must have a 10-pound ABC-rated fire extinguisher. <u>1,2,3,4</u> Obtain fire extinguisher use training. <u>1,2,3,4</u> Obtain "hot work" permits when appropriate. <u>1,2,3,4</u> Do not smoke or operate spark-ignition equipment within 50 feet of explosive or flammable storage or where flammable liquid or vapor is present. <u>1,2,3,4</u> Do not use equipment that may generate a spark where the potential presence of explosive gas or vapor is suspected. <u>1,2</u> Use a combustible gas indicator when working at a site with the potential for explosive gas or when the potential for flammable vapor exists. <u>1,2</u> Use fans as an engineering control to limit buildup of explosive gas.

5.4 Required Personal Protective Equipment and Related Safety Equipment

This section describes the available levels of PPE as specified by 29 CFR 1910.120, Appendix B. PPE for Clear Creek employees will be supplied by Clear Creek. Table 4 presents the project-specific PPE by work activity.

Subcontractors and site visitors will be required to supply their own PPE.

5.4.1 Levels of Personal Protective Equipment

Level D PPE

A work uniform affording minimal protection used for nuisance contamination only. The following constitute Level D PPE, which may be used as appropriate:

- Coveralls or field clothing
- Gloves (as applicable)
- Boots/shoes; chemical-resistant, steel toe and shank
- Safety glasses or chemical splash goggles
- Hardhat (as applicable)
- Earplugs and/or earmuffs (as applicable)
- Escape mask (as applicable)
- Face shield (as applicable)

Modified Level D PPE

This level of protection applies when concentration(s) and type(s) of airborne substance(s) are known to be below the PELs/TLVs. Air purifying respirators should be readily available. The following constitute Modified Level D PPE, which may be used as appropriate:

- Chemical-resistant clothing
- Coveralls or field clothing
- Gloves, outer, chemical-resistant
- Gloves, inner, chemical-resistant
- Boots, chemical-resistant, steel toe and shank
- Boot covers, chemical-resistant (disposable)
- Safety glasses or chemical splash goggles
- Hardhat (as applicable)
- Earplugs and/or earmuffs (as applicable)
- Escape mask (as applicable)
- Face shield (as applicable)

Note: Use of an air-purifying respirator with Modified Level D PPE will constitute Level C PPE.

Level C PPE

This level of protection applies when the concentration(s) and type(s) of airborne substance(s) are known and the criteria for using air purifying respirators are met. The following constitute Level C PPE, which may be used as appropriate:

- Full-face or half-mask, air purifying respirators (NIOSH-approved) with appropriate cartridges. Employees working on this project will use organic vapor/acid mist/HEPA filter cartridges.
- Chemical-resistant clothing (hooded unless approved by the DSHO)
- Coveralls or field clothing
- Gloves, outer, chemical-resistant
- Gloves, inner, chemical-resistant
- Boots, chemical-resistant, steel toe and shank
- Boot covers, chemical-resistant (disposable)
- Hardhat (as applicable)

- Earplugs and/or earmuffs (as applicable)
- Face shield (as applicable)

5.4.2 Unknown Situations

For unknown, uncharacterized, and unanticipated situations, field activities must begin in Level B PPE. Downgrade to Level C or D PPE will not be permitted until analytical data for the site have been reviewed. The DSHO and/or the SSHO must approve the downgrade.

5.5 Air Monitoring for Project Operations

This section describes instruments and procedures that can be used for air monitoring activities. It may not be necessary to perform all of these activities at every project site. Final decisions regarding air monitoring will be made by the DSHO or the SSHO.

A daily monitoring log will be kept by the SSHO for each piece of air monitoring equipment. The following information will be recorded:

- Name and model number of the equipment
- Calibration information
- Field work to be performed
- Air monitoring results and monitoring locations
- Accidents or incidents
- Unusual occurrences and personnel complaints
- Weather information
- Post calibration results, if performed

Air monitoring results will also be recorded in the field logbook.

5.5.1 Gases and Vapors

A photoionization detector (PID) or a flame ionization detector (FID) will be used to monitor breathing zone concentrations of volatile organic compounds (VOCs). Monitoring will be conducted continuously during sampling or intrusive activities. A sustained reading of 1 ppm for a period of at least one minute in the breathing zone will prompt an immediate work stoppage until a quantitative determination of VOC can be determined. Calibration of monitoring equipment will be performed daily before startup of work. Calibration gas to be used will be specific to the instrument per manufacturer instructions.

Action levels for known contaminants shall be based on the PEL or TLVs of the contaminants, whichever level is the most conservative. Action levels for unidentified total atmospheric organic contaminants are based on the following:

Instrument Reading for One Minute	Action
Background	Level D
Above background	Level D - introduce engineering controls (e.g., fans)
150 ppm above background	Level C

5.5.2 Explosion Hazard

A combustible gas indicator (CGI) will be used at sites as appropriate to monitor the possible presence of explosive gases. Equipment calibration will be performed daily before startup of work per manufacturer instructions. The alarm will be set to 10 percent of the LEL. If feasible, calibration gas to be used will be specific to the combustible gases suspected to be present.

Continuous monitoring for the presence of combustible gases will be performed at the sampling point. If the monitoring instrument indicates the LEL is greater than 10 percent, personnel must leave the area. Fans should be used to lower the LEL. Personnel must not reenter the area until the LEL is less than 10 percent.

5.5.3 Oxygen Deficiency in Confined Spaces

Before entering a confined space, an oxygen meter must be used to measure the oxygen concentration in air. If the oxygen concentration is less than 19.5 percent or greater than 23.5 percent, entry to the space is prohibited. Fans should be used to ventilate the area. If the oxygen concentration cannot be stabilized between 19.5 and 23.5 percent, Level B PPE must be donned to enter the confined space. (Note: Entry into a confined space may require that a permit be obtained from the DSHO. Further information on confined space entry is presented in Section 5.3.)

5.6 Hazardous Pathways and Engineering Controls

Potential hazardous pathways at the site may include organic vapor, dust, and contact with petroleum hydrocarbon impacted soil or water. Utilize a respirator and/or fans if organic vapors measurements, as recorded by the PID, exceed the levels stated in Section 5.5.1. Use Nitrile gloves if contact with impacted soil or water is necessary.

6.0 DECONTAMINATION PROCEDURES

Procedures to be followed for equipment and personnel decontamination and disposal of investigation-derived material are described below.

6.1 Equipment Decontamination

Equipment that comes in contact with potentially contaminated soil, water, or air containing dusts or other aerosols, including equipment used for soil sampling and excavation, will be cleaned before and after each use on this project. Decontamination will consist of steam cleaning and/or detergent (Liqui-Nox™ or equivalent) wash, with a water rinse.

6.2 Personnel Decontamination

The sequence for personnel decontamination for Level C PPE field activities is described below. Personnel decontamination for Level D PPE or Modified Level D PPE activities will include the applicable procedures described below. Decontamination will occur as follows:

- If gross contamination is present, wash PPE in detergent or other appropriate solution rinse in clean water.
- Remove disposable overboots (if used). Remove outer gloves.
- Wash chemical-resistant boots with detergent solution and rinse with clean water.
- Remove coveralls. Starting at the neck, roll the coveralls off from the inside out and down past the boots. Take care to prevent the release and dispersion of dusts or prevent contact with decontamination water that may have accumulated on the coveralls. Do not contaminate clothing inside the coveralls during removal.
- Remove the respirator. Clean and disinfect the respirators and place into a plastic bag for storage.
- Place disposable PPE in an appropriate container for disposal.
- Remove liner gloves.
- Thoroughly wash hands and face.

Each drum will be issued a unique number. The number will be recorded on the drum and in a log. Information as to the drum contents, the location the contents were collected, and the date filled will also be recorded on the drum and in the drum log.

7.0 EMERGENCY PROCEDURES

Pertinent emergency information and the contingency plan are provided in this section.

7.1 Emergency Telephone Numbers

Ambulance: 911
Police: 911
Fire Department: 911
Urgent Care (480) 814-1560
Hospital: (480) 728-3211 Emergency
Name: Chandler Regional Hospital
Emergency Air Lift: 911
Poison Control Center, local: (800) 362-0101
CHEMTREC: 1 (800) 424-9300

Client Contact:	<u>Chris Alger</u>	Office: <u>(510) 834-4747</u>	Other: <u>(510) 338-2794</u>
Project Manager:	<u>Tom Suriano</u>	Office: <u>(480) 695-7131</u>	Other: <u>(602) 762-0996</u>
DSHO:	<u>Dave Giles</u>	Office: <u>(480) 695-7131</u>	Other: <u>(602) 762-0676</u>

7.2 How to Report an Emergency

When calling for assistance in an emergency situation, the following minimum information should be provided:

1. Name of person calling
2. Telephone number of caller's location
3. Name of person(s) exposed or injured
4. Nature of emergency
5. Actions already taken

The recipient of the call should hang up first - not the caller.

7.3 Emergency Routes

Hospital: Chandler Regional Hospital

Telephone Number: (480) 728-3211 Emergency
Address: 475 S. Dobson Rd.
Chandler, AZ 85224

Route to Hospital:

Leave site heading EAST (left) on Allison Road, to Kyrene Rd. (~0.67 mi.). Take Kyrene Road NORTH (left) to Chandler Blvd. (~ 1.2 mi.). Take Chandler Blvd. EAST (right) to Dobson Road (~4 mi.). Take Dobson Road SOUTH (right) to Chandler Regional Hospital ER – for a LIFE THREATENING injury OR NextCare Urgent Care Center – for NON-LIFE THREATENING Injury.

The following hand/body emergency communication signals should be used when other forms of communication are difficult or impossible:

Signal	Meaning
Hand clutching throat	Out of air/can't breathe
Hands on top of head	Need assistance

Thumbs up	OK/I'm all right/I understand
Grip partner's wrist or both hands around partner's waist	Leave area immediately
If the emergency occurs in the Exclusion Zone, all field personnel will quickly move to the Contamination Reduction Zone for an appropriate decontamination before exiting to the Support Zone. In life-threatening emergencies, decontamination may not be appropriate. The emergency decontamination decision will be made by the SSHO. Emergency situations occurring outside of the Exclusion Zone in Level D PPE will not require decontamination at the Contamination Reduction Zone before administering first aid.	

Minor emergencies will be handled utilizing the onsite first-aid kit. A portable emergency eyewash or 32 ounces of eyewash fluid will be available in the field vehicle. Either an emergency shower or a shower facility will be available for personnel decontamination. If working at a remote location (more than 15 minutes from an emergency medical facility), at least one onsite Clear Creek person will be trained in first aid. The person trained in first aid will be identified during each tailgate session. Appendix D is a summary of American National Red Cross first-aid procedures. The appropriate emergency response personnel (i.e., ambulance and fire department) will be contacted for all major emergencies.

Routes to the nearest hospital are provided on Figure 1, and described in Section 7.3. A written report of all emergencies will be submitted to Dave Giles. Accident forms are located in Attachment C. Copies of this report will also be sent to the appropriate agencies.

7.5 Contingency Plan

This Contingency Plan has been developed by CCA to present procedures that should be followed in the event of an emergency at a field operation. A variety of events that are potential hazards to human health and the environment are discussed, including the following:

- A funnel cloud or tornado sighting
- An explosion
- A chemical or petroleum spill or accident
- Other events presenting a hazard to human health or the environment

This section also specifies the general procedures you should follow, who you should notify, and the information you should report if you are the first on the scene of an emergency.

7.5.1 Response Sequence for First Arrivals

If you are first on the scene, respond as follows:

- Evacuate the incident area (if necessary). Remember that your safety must be the primary consideration.
- Restrict access to the incident area.
- Restrict the use of ignition sources for incidents involving flammable substances.
- Call the Site Supervisor or the local emergency response organization. Report the following information:
 - Your name
 - Company affiliation
 - Telephone number from which you are calling
 - Location and type of incident
 - Injuries, if any, and the number and type of those injuries
 - Details concerning the substance(s) involved (identification, amount, spill rate, size of area involved), if known

- Direction the spill is moving and the direction the wind may be dispersing airborne contaminants
 - Surficial material on which the spill occurred (i.e., asphalt, gravel, etc.)
 - Any first response action that has been taken
 - The time the incident occurred or when you discovered it
 - Any additional pertinent information
- Notify the SSHO after the emergency response team has been contacted. The SSHO will then notify the local DSHO.
- Coordinate with emergency response personnel when they arrive.

7.5.2 Response for Incidents Involving Another Contractor

If the incident involves another contractor's activity:

- Evacuate the area immediately.
- Decontaminate and remove PPE if the incident is not life- or health-threatening.
- Proceed to the predetermined assembly point.
- Call for assistance.
- Make sure the SSHO knows you are present.

7.5.3 Emergency Response for Severe Weather Conditions

This section specifies what you should do in the event of a severe weather emergency, including electrical storms, high winds, heavy rain or hail, and tornadoes.

Electrical Storms

- Seek shelter at the support facility or in the field vehicles.
- Do not stand near or under high objects, such as trees and drilling rigs.
- If possible, lower the drilling rig mast.

High Winds

- Seek shelter at the support facility (if anchored) or in the field vehicles.
- Do not drive high-profile vehicles at high speeds.
- Park vehicles heading into the wind.
- Don a respirator or wear safety goggles and a kerchief covering your nose and mouth.

Heavy Rain or Hail

- Seek shelter at the support facility or in the field vehicles.
- Do not attempt to drive a vehicle if you are in an area that is or has the potential for flooding unless you are moving out of a low area.

Tornadoes

- Seek shelter underground or in a closet, bathroom, or interior wall of a substantial building. Get under something sturdy and cover your head.
- Do not stay in a trailer or vehicle. Leave the trailer or vehicle and lie flat in the nearest ditch if substantial shelter is not available.
- Stay away from large areas of glass.
- Stay away from large unsupported roofs.

7.5.5 Emergency Response for Flash Floods

If a flash flood warning is issued, climb to higher ground. Seek shelter on stable ground. Do not stay in an area that is characterized by uncompacted material on a steep slope.

7.5.6 Emergency Response for Fires

If a small fire occurs, extinguish it with the fire extinguisher in the field vehicle. Remember to follow these directions to put out the fire:

- Aim at the base of the flame.
- Use the appropriate type of fire extinguisher (e.g., do not use a water type fire extinguisher or an electrical fire).
- Remember that the spray only lasts a few seconds.
- If a large fire occurs at the work site, follow these instructions:
 - Move flammable and combustible items out of the path of the fire if such action can be performed safely.
 - Call the fire department and report the information as shown in Section 7.1 and 7.2.
 - Do not attempt to put out a large fire with the field vehicle fire extinguisher.
 - Report the incident to the Site Supervisor.

7.5.7 Fire Prevention

Steps to be taken to minimize the potential of a fire include the following:

- Obey "No Smoking" signs.
- Shut off engines before refueling, if possible.
- Use intrinsically safe equipment in areas potentially containing flammable vapor.

7.5.8 Emergency Response for Explosions

If an explosion occurs, follow these instructions:

- Evacuate the site immediately.
- Do not address medical emergencies until you are out of danger.
- Call the Site Supervisor or local emergency response organization when you are out of danger to report the incident. Report the information as shown in Section 7.1 and 7.2.

7.5.9 Emergency Response for Spills

The following sections provide guidance regarding emergency response to an accidental discharge of gasoline, including initial response to the incident and cleanup.

7.5.10 Initial Spill Response

When a spill occurs:

- Minimize or contain the flow by shutting off a valve, repairing the leak, righting an overturned barrel, or whatever action is appropriate. Remember that your safety is of primary concern. Only attempt emergency response actions if you can do so without injury or harm to yourself.
- Provide first aid to injured persons as needed.

- If the spill occurs on a porous surface (e.g., soil, gravel) mark the area in preparation for excavation if that is determined to be the appropriate response action. If the spill occurs on concrete, asphalt, or similar material, use sorbent material to contain the spill. Sorbent materials will be kept in field vehicles and in the support facility. Cover the area with soil, a tarp, plastic, or other appropriate material if the spilled material is volatile and cannot be cleaned up immediately.
- Call the Site Supervisor or the local emergency response organization (as applicable) to report the incident. Supply the information as shown in Section 7.1 and 7.2. At least two CCA personnel must remain onsite in a safe location 30 feet upwind of the spill until emergency response representatives arrive.
- Dependent on the location and chemical nature of the spilled liquid, initial response may require donning Level C or Level B PPE.

7.5.11 Spill Site Decontamination

Clear Creek and subcontractor site personnel involved in the response action will undergo limited personal decontamination upwind of the incident site. Further decontamination will be completed at the decontamination trailer in the support zone. The SSHO will authorize site personnel to leave the job site or continue work, as appropriate. The Site Supervisor will provide guidance regarding decontamination and/or disposition of equipment and vehicles.

- If personnel come in contact with fuel or site contaminants, they should remove and dispose contaminated PPE, change out of contaminated field clothing, and wash exposed skin with soap and water.

7.5.12 Spill Prevention

To minimize the potential for a spill, you should follow these guidelines:

- Receive instruction concerning recognition of potential spill problems, preventative maintenance actions, and increased safety awareness.
- Inspect stored materials at the beginning of each work shift. The container condition, as well as any notation of leaks or staining that may be related to or indication of a potential spill, will be recorded in a field logbook. Deficiencies or corrective actions must be reported immediately to the Site Supervisor.
- Inspect transfer vehicles and equipment at the beginning of each day. Equipment conditions, as well as any notation of leaks or staining that may be related to or indicative of a potential spill, will be recorded in a field logbook. Loose and or worn connections and worn hoses will also be noted. Any abnormalities must be reported immediately to the Site Supervisor and steps should be taken to remedy the situation before continuing transfer activities.
- Make sure materials being stored are compatible with the containers in which they are being stored. Incompatible materials will not be stored together. Acids and bases will be stored in separate cabinets affixed with caution labels. Spillable items, if stored on shelves, will be no higher than 4 feet off the floor surface. (This height is to limit the potential for getting a toxic substance in the eyes).
- Store containers larger than 1 gallon separately from smaller quantities. Larger liquid storage containers will be stored in a warehouse and must have a secondary containment system. This system can be as simple as a liner with a berm constructed of 4-inch by 4-inch boarding and should be able to contain an amount 10 percent greater than that of the original container.
- Transfer liquid with catch basins under each joint or valve or with the hose or pipe lined so that no liquid can escape.

7.5.13 Cleanup Materials and Used Personal Protective Equipment Disposal

- Materials used in spill cleanup must be collected and placed in barrels and stored at the job site. Contaminated soil, soil cleaned from equipment, and decontamination water will also be drummed and

stored. The Site Supervisor should assess whether the surficial material on which the spill is located requires treatment or removal and relay this information to the project manager.

- Decontamination of large equipment may be performed at a temporary decontamination pad set up at the site or at a central decontamination pad.
- Tools used during spill cleanup will be thoroughly cleaned at the decontamination pad.
- Used PPE will be placed in barrels and stored at the job site.

7.5.14 Responsibilities of Site Personnel

Clear Creek 's Responsibilities

- Follow the first response directives shown in Section 7.1 and 7.2.
- Wear the correct and appropriate PPE for task completion.
- Use monitoring equipment applicable to the anticipated hazards (i.e., CGI, FID, PID, etc.)
- Have a decontamination area set up for fieldwork as appropriate.
- Use approved decontamination procedures.
- Treat minor injuries using the onsite first-aid kit.
- Take personnel with serious injuries to Mesa General Hospital or contact a medical emergency response team.
- Contact emergency response for health- or life-threatening injuries. Victims should be taken to Mesa General Hospital by the medical emergency response team.
- Perform remedial actions as appropriate.

7.5.15 Emergency Response Equipment

The following is a list of minimum equipment that is required to be available for emergency response actions:

- 5-pound ABC-rated fire extinguisher
- First-aid kit
- Eyewash station or eyewash bottles (totaling 32 ounces)

The emergency response equipment will be located in a designated location and/or Clear Creek Vehicle. First aid kits and fire extinguishers will be located in the vicinity of the drill rig and in the Clear Creek vehicle.

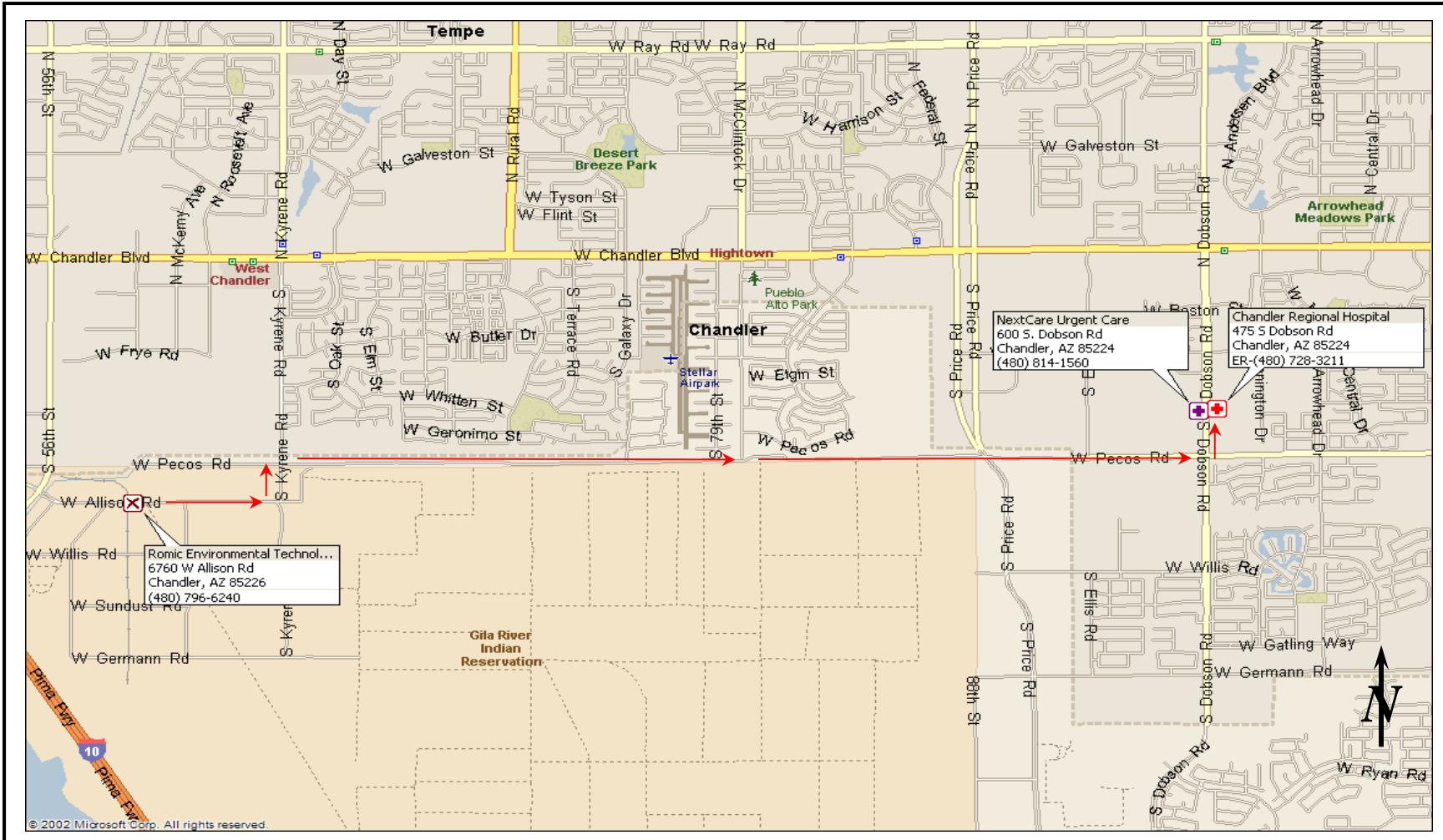
8.0 GLOSSARY

>	Greater than
<	Less than
°C	Degrees Celsius
°F	Degrees Fahrenheit
µg/m ³	Micrograms per cubic meter
µg/g	Micrograms per gram
µg/l	Micrograms per liter
ACGIH	American Conference of Governmental Industrial Hygienists
bgs	Below ground surface
BTEX	Benzene, toluene, ethylene, xylene
CFR	Code of Federal Regulations
CGI	Combustible gas indicator
cm ³	Cubic centimeter
CPR	Cardiopulmonary resuscitation
CRZ	Contamination reduction zone
DOT	U.S. Department of Transportation
DSHO	Designated Safety and Health Officer
EPA	U.S. Environmental Protection Agency
FID	Flame ionization detector
g	Gram
g/ml	Grams per milliliter
hsa	Hollow stem auger
HEPA	High-efficiency particulate/air
IDLH	Immediately dangerous to life and health
LEL	Lower explosive limit
mg/g	Milligrams per gram
mg/l	Milligrams per liter
mm	Millimeter
mm ³	Cubic millimeters
mph	Miles per hour
N/A	Not applicable
N/F	Nonflammable
N/I	No information available
NIOSH	National Institute for Occupational Safety and Health
OSHA	Occupational Safety and Health Administration
PCB	Polychlorinated biphenyl
PEL	Permissible exposure level
PID	Photoionization detector
ppb	Parts per billion
PPE	Personal protective equipment
ppm	Parts per million
RI/FS	Remedial investigation/feasibility study
SCBA	Self-contained breathing apparatus
SSHO	Site Safety and Health Officer
HASP	Health and Safety Plan
TLV	Threshold Limit Value
TOMES	Toxicology, Occupational Medicine, and Environmental Series
TPH	Total petroleum hydrocarbons

TWA Time weighted average
UEL Upper explosive limit



FIGURES



Leave site heading WEST on Allison Road, to Kyrene Rd.
(~0.67 mi.). Take Kyrene Road NORTH (left) to Pecos Road
(~ 0.125 mi.). Take Pecos Road EAST (right) to Dobson Road
(~4 mi.). Take Dobson Road NORTH to Chandler Regional
Hospital ER – for LIFE THREATENING injury OR NextCare
Urgent Care Center – for NON-LIFE THREATENING Injury.

FIGURE 1
HOSPITAL ROUTE MAP
RCRA Facility Investigation (RFI)
Health and Safety Plan
Romic Environmental Technologies
Gila River Indian Community, Arizona

TABLES

TABLE 1
IDENTIFIED CHEMICAL COMPOUNDS

NOTE: Delete/add chemical(s) as needed

CHEMICAL	CONCENTRATION OF RECENT MAXIMUM DETECTION
Trichloroethene	36 µg/L
Tetrachloroethene	65 µg/L
1,1-DCE	6.6 µg/L
cis-1,2-DCE	1.7 µg/L
1,1 DCA	1.1 µg/L
1,2 DCA	8.5 µg/L
1,1,2 TCA	1.2 µg/L

NOTES:

µg/L micrograms per liter

All chemical concentrations were detected in groundwater samples.

TABLE 2
HAZARD ANALYSIS

Job Project by Activity Number

Hazards	Activity 1	Activity 2	Activity 3	Activity 4
Physical	hand tools slip/trip/fall lifting	hand tools slip/trip/fall lifting	hand tools slip/trip/fall lifting	hand tools slip/trip/fall lifting
Mechanical	heavy equipment	heavy equipment	heavy equipment	heavy equipment
Trenching/Excavating	N/A	N/A	N/A	N/A
Electrical	Buried underground utilities	N/A	N/A	Portable generator, pump connections
Chemical	chemicals in vapors, dust and water	chemicals in vapor, dust and water	chemicals in vapors & water	chemicals in vapors & water
Asbestos	N/A	N/A	N/A	N/A
Temperature	heat stress, cold stress			
Acoustical	high noise levels	high noise levels	high noise levels	high noise levels
Confined Spaces	N/A	N/A	N/A	N/A
Natural	high winds, weather	high winds, weather	high winds, weather	high winds, weather
Biohazards	plants, insects, animals, reptiles			
Fire/Explosion	flammable chemicals released to air			

N/A Not Applicable

Activity 1 Drilling and Soil Sampling

Activity 2 Well Installation

Activity 3 Water Levels

Activity 4 Groundwater Sampling

TABLE 3
RISK ASSESSMENT
Page 1 of 2

	Hazard	Exposure ^a	Probability ^b	Consequence ^c
Physical	hand tools	frequent	unusual	minor to serious
	slip/trip/fall	continuous	likely	minor to fatal
	lifting	frequent	unusual	minor to moderate
Mechanical	heavy equipment	continuous	likely	minor to fatal
Trenching/Excavation	N/A	N/A	N/A	N/A
Electrical	power lines	frequent	improbable to likely	moderate to fatal
	generator	frequent	improbable to likely	moderate to fatal
Chemical	chemicals in air	continuous	unusual	minor to chronic
	chemicals in water	continuous	improbable	minor to chronic
	dust	continuous	unusual	minor to chronic
Asbestos	N/A	N/A	N/A	N/A
Temperature	heat stress	continuous	likely	minor to fatal
	cold stress	continuous	likely	minor to fatal
Acoustical	drilling rig,	continuous	Unusual	chronic
	earthmoving equipment	continuous	Unusual	chronic
	generator	continuous	unusual	chronic
Confined Spaces	N/A	N/A	N/A	N/A
Natural	sunlight	continuous	likely	minor to serious
	storms	occasional	unusual	minor to fatal
Biohazards	plants	seldom	improbable	minor to moderate
	insects	continuous	likely	minor to fatal
	animals	seldom	improbable	minor to fatal
	reptiles	frequent	unusual	minor to fatal
Fire/Explosion	flammable chemicals released to air	frequent	unusual	minor to fatal

N/A Not Applicable

^a Exposure: the frequency of exposure to the hazardous event

Continuous = many times daily

Frequent = once/day or twice/day

Occasional = once/week to once/month

TABLE 3
RISK ASSESSMENT
Page 2 of 2

^b Probability: the likelihood that an injury will occur after exposure to the hazardous event

Certain = certain or almost certain

Likely = not unusual, 50 percent chance of occurring

Unusual = would happen less often than not

Improbable = not likely to happen

^c Consequence: the degree of injury resulting from exposure to the hazardous event if injury occurs

Fatal = fatality

Serious = serious injury, including chemical exposure requiring hospitalization

Moderate = moderate injury, including chemical exposure requiring outpatient medical treatment

Minor = minor injury, including chemical exposure requiring onsite first aid

Chronic = chemical acoustical, or other exposure above the Threshold Limit Value (TLV) or other recommended standard that may not produce immediate acute effects (especially chronic toxicants)

TABLE 4
TASK-SPECIFIC PERSONAL PROTECTIVE EQUIPMENT

Level D	Modified D	Level C
Head Hardhat	Head Hardhat	Head Hardhat
Eye/Face Safety glasses Chemical goggles Face shield	Eye/Face Safety glasses Chemical goggles Face shield	Eye/Face Safety glasses Chemical goggles Face shield
Ear Ear plugs Earmuffs	Ear Ear plugs Earmuffs	Ear Ear plugs Earmuffs
Hand (Underglove) N-Dex [®] Thermax [®]	Hand (Underglove) N-Dex [®] Thermax [®]	Hand (Underglove) N-Dex [®] Thermax [®]
Body Rainsuit Cloth coveralls	Hand (Outerglove) Nitrile Neoprene Butyl PVC Viton	Hand (Outerglove) Nitrile Neoprene Butyl PVC Viton SilverShield [®]
Foot STCR boot	Body Tyvek [®] Polycoated Tyvek [®] Foot STCR boot Overboot	Body Tyvek [®] Polycoated Tyvek [®] Foot STCR boot Overboot
		Face/respiratory Full-face PAPR Full-face respirator Half-face respirator Cartridges (specify type)

PAPR Powered Air Purifying Respirator
 PVC Polyvinyl chloride
 SCBA Self-contained breathing apparatus
 STCR Steel-toed, chemical-resistant

* With 5 minute egress bottle.

ATTACHMENT A

HAZARDOUS PROPERTY INFORMATION



NIOSH Pocket Guide to Chemical Hazards

Tetrachloroethylene		CAS 127-18-4
<chem>Cl2C=CCl2</chem>		RTECS KX3850000
Synonyms & Trade Names Perchlorethylene, Perchloroethylene, Perk, Tetrachlorethylene		DOT ID & Guide 1897 160
Exposure Limits NIOSH REL: Ca Minimize workplace exposure concentrations. See Appendix A OSHA PEL†: TWA 100 ppm C 200 ppm 300 ppm (5-minute maximum peak in any 3-hours)		
IDLH Ca [150 ppm] See: 127184		Conversion 1 ppm = 6.78 mg/m ³
Physical Description Colorless liquid with a mild, chloroform-like odor.		
MW: 165.8	BP: 250°F	FRZ: -2°F
VP: 14 mmHg	IP: 9.32 eV	Sp.Gr: 1.62
Fl.P: NA	UEL: NA	LEL: NA
Noncombustible Liquid, but decomposes in a fire to hydrogen chloride and phosgene.		
Incompatibilities & Reactivities Strong oxidizers; chemically-active metals such as lithium, beryllium & barium; caustic soda; sodium hydroxide; potash		
Measurement Methods NIOSH 1003 ; OSHA 1001 See: NMAM or OSHA Methods		
Personal Protection & Sanitation (See protection) Skin: Prevent skin contact Eyes: Prevent eye contact Wash skin: When contaminated Remove: When wet or contaminated Change: No recommendation Provide: Eyewash, Quick drench		First Aid (See procedures) Eye: Irrigate immediately Skin: Soap wash promptly Breathing: Respiratory support Swallow: Medical attention immediately
Important additional information about respirator selection Respirator Recommendations NIOSH At concentrations above the NIOSH REL, or where there is no REL, at any detectable concentration: (APF = 10,000) Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode (APF = 10,000) Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained positive-pressure breathing apparatus Escape: (APF = 50) Any air-purifying, full-facepiece respirator (gas mask) with a chin-style, front- or back-mounted organic vapor canister/Any appropriate escape-type, self-contained breathing apparatus		
Exposure Routes inhalation, skin absorption, ingestion, skin and/or eye contact		
Symptoms Irritation eyes, skin, nose, throat, respiratory system; nausea; flush face, neck; dizziness, incoordination; headache, drowsiness; skin erythema (skin redness); liver damage; [potential occupational carcinogen]		
Target Organs Eyes, skin, respiratory system, liver, kidneys, central nervous system		
Cancer Site [in animals: liver tumors]		
See also: INTRODUCTION See ICSC CARD: 0076 See MEDICAL TESTS: 0179		



NIOSH Pocket Guide to Chemical Hazards

Trichloroethylene		CAS 79-01-6
<chem>CICH=CCl2</chem>		RTECS KX4550000
Synonyms & Trade Names Ethylene trichloride, TCE, Trichloroethene, Trilene		DOT ID & Guide 1710 160
Exposure Limits NIOSH REL: Ca See Appendix A See Appendix C OSHA PEL†: TWA 100 ppm C 200 ppm 300 ppm (5-minute maximum peak in any 2 hours)		
IDLH Ca [1000 ppm] See: 79016		Conversion 1 ppm = 5.37 mg/m ³
Physical Description Colorless liquid (unless dyed blue) with a chloroform-like odor.		
MW: 131.4	BP: 189°F	FRZ: -99°F
VP: 58 mmHg	IP: 9.45 eV	Sp.Gr: 1.46
Fl.P: ?	UEL(77°F): 10.5%	LEL(77°F): 8%
Combustible Liquid, but burns with difficulty.		
Incompatibilities & Reactivities Strong caustics & alkalis; chemically-active metals (such as barium, lithium, sodium, magnesium, titanium & beryllium)		
Measurement Methods NIOSH 1022 , 3800 ; OSHA 1001 See: NMAM or OSHA Methods		
Personal Protection & Sanitation (See protection) Skin: Prevent skin contact Eyes: Prevent eye contact Wash skin: When contaminated Remove: When wet or contaminated Change: No recommendation Provide: Eyewash, Quick drench		First Aid (See procedures) Eye: Irrigate immediately Skin: Soap wash promptly Breathing: Respiratory support Swallow: Medical attention immediately
Important additional information about respirator selection Respirator Recommendations NIOSH At concentrations above the NIOSH REL, or where there is no REL, at any detectable concentration: (APF = 10,000) Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode (APF = 10,000) Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained positive-pressure breathing apparatus Escape: (APF = 50) Any air-purifying, full-facepiece respirator (gas mask) with a chin-style, front- or back-mounted organic vapor canister/Any appropriate escape-type, self-contained breathing apparatus		
Exposure Routes inhalation, skin absorption, ingestion, skin and/or eye contact		
Symptoms Irritation eyes, skin; headache, visual disturbance, lassitude (weakness, exhaustion), dizziness, tremor, drowsiness, nausea, vomiting; dermatitis; cardiac arrhythmias, paresthesia; liver injury; [potential occupational carcinogen]		
Target Organs Eyes, skin, respiratory system, heart, liver, kidneys, central nervous system		
Cancer Site [in animals: liver & kidney cancer]		
See also: INTRODUCTION See ICSC CARD: 0081 See MEDICAL TESTS: 0236		



NIOSH Pocket Guide to Chemical Hazards

Vinylidene chloride		CAS 75-35-4
<chem>CH2=CCl2</chem>		RTECS KV9275000
Synonyms & Trade Names 1,1-DCE; 1,1-Dichloroethylene; 1,1-Dichloroethylene; VDC; Vinylidene chloride monomer; Vinylidene dichloride		DOT ID & Guide 1303 130P (inhibited)
Exposure Limits	NIOSH REL: Ca See Appendix A	
	OSHA PEL†: none	
IDLH Ca [N.D.] See: IDLH INDEX		Conversion
Physical Description Colorless liquid or gas (above 89°F) with a mild, sweet, chloroform-like odor.		
MW: 96.9	BP: 89°F	FRZ: -189°F
VP: 500 mmHg	IP: 10.00 eV	Sp.Gr: 1.21
Fl.P: -2°F	UEL: 15.5%	LEL: 6.5%
Class IA Flammable Liquid: Fl.P. below 73°F and BP below 100°F.		
Incompatibilities & Reactivities Aluminum, sunlight, air, copper, heat [Note: Polymerization may occur if exposed to oxidizers, chlorosulfonic acid, nitric acid, or oleum. Inhibitors such as the monomethyl ether of hydroquinone are added to prevent polymerization.]		
Measurement Methods NIOSH 1015 ; OSHA 19 See: NMAM or OSHA Methods		
Personal Protection & Sanitation (See protection) Skin: Prevent skin contact Eyes: Prevent eye contact Wash skin: When contaminated Remove: When wet (flammable) Change: No recommendation Provide: Eyewash, Quick drench		First Aid (See procedures) Eye: Irrigate immediately Skin: Soap flush immediately Breathing: Respiratory support Swallow: Medical attention immediately
Important additional information about respirator selection Respirator Recommendations NIOSH At concentrations above the NIOSH REL, or where there is no REL, at any detectable concentration: (APF = 10,000) Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode (APF = 10,000) Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained positive-pressure breathing apparatus Escape: (APF = 50) Any air-purifying, full-facepiece respirator (gas mask) with a chin-style, front- or back-mounted organic vapor canister/Any appropriate escape-type, self-contained breathing apparatus		
Exposure Routes inhalation, skin absorption, ingestion, skin and/or eye contact		
Symptoms Irritation eyes, skin, throat; dizziness, headache, nausea, dyspnea (breathing difficulty); liver, kidney disturbance; pneumonitis; [potential occupational carcinogen]		
Target Organs Eyes, skin, respiratory system, central nervous system, liver, kidneys		
Cancer Site [in animals: liver & kidney tumors]		
See also: INTRODUCTION See ICSC CARD: 0083		



NIOSH Pocket Guide to Chemical Hazards

Dichloroacetylene		CAS 7572-29-4
<chem>C2Cl2</chem>		RTECS AP1080000
Synonyms & Trade Names DCA, Dichloroethyne [Note: DCA is a possible decomposition product of trichloroethylene or trichloroethane.]		DOT ID & Guide
Exposure Limits NIOSH REL: Ca C 0.1 ppm (0.4 mg/m ³) See Appendix A OSHA PEL†: none		
IDLH Ca [N.D.] See: IDLH INDEX		Conversion 1 ppm = 3.88 mg/m ³
Physical Description Volatile oil with a disagreeable, sweetish odor. [Note: A gas above 90°F. DCA is not produced commercially.]		
MW: 94.9	BP: 90°F (Explodes)	FRZ: -58 to -87°F
VP: ?	IP: ?	Sp.Gr: 1.26
Fl.P: ?	UEL: ?	LEL: ?
Combustible Liquid		
Incompatibilities & Reactivities Oxidizers, heat, shock		
Measurement Methods None available See: NMAM or OSHA Methods		
Personal Protection & Sanitation (See protection) Skin: Prevent skin contact Eyes: Prevent eye contact Wash skin: When contaminated Remove: When wet (flammable) Change: No recommendation Provide: Eyewash, Quick drench		First Aid (See procedures) Eye: Irrigate immediately Skin: Soap flush immediately Breathing: Respiratory support Swallow: Medical attention immediately
Important additional information about respirator selection Respirator Recommendations NIOSH At concentrations above the NIOSH REL, or where there is no REL, at any detectable concentration: (APF = 10,000) Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode (APF = 10,000) Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained positive-pressure breathing apparatus Escape: (APF = 50) Any air-purifying, full-facepiece respirator (gas mask) with a chin-style, front- or back-mounted organic vapor canister/Any appropriate escape-type, self-contained breathing apparatus		
Exposure Routes inhalation, skin absorption, ingestion, skin and/or eye contact		
Symptoms Headache, loss of appetite, nausea, vomiting, intense jaw pain, cranial nerve palsy; in animals: kidney, liver, brain injury; weight loss; [potential occupational carcinogen]		
Target Organs central nervous system		
Cancer Site [in animals: kidney tumors]		
See also: INTRODUCTION See ICSC CARD: 1426		



NIOSH Pocket Guide to Chemical Hazards

1,2-Dichloroethylene		CAS 540-59-0
CICH=CHCl		RTECS KV9360000
Synonyms & Trade Names Acetylene dichloride, cis-Acetylene dichloride, trans-Acetylene dichloride, sym-Dichloroethylene		DOT ID & Guide 1150 130P
Exposure Limits	NIOSH REL: TWA 200 ppm (790 mg/m ³)	
	OSHA PEL: TWA 200 ppm (790 mg/m ³)	
IDLH 1000 ppm See: 540590	Conversion 1 ppm = 3.97 mg/m ³	
Physical Description Colorless liquid (usually a mixture of the cis & trans isomers) with a slightly acrid, chloroform-like odor.		
MW: 97.0	BP: 118-140°F	FRZ: -57 to -115°F
VP: 180-265 mmHg	IP: 9.65 eV	Sp.Gr(77°F): 1.27
Fl.P: 36-39°F	UEL: 12.8%	LEL: 5.6%
Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.		
Incompatibilities & Reactivities Strong oxidizers, strong alkalis, potassium hydroxide, copper [Note: Usually contains inhibitors to prevent polymerization.]		
Measurement Methods NIOSH 1003 ; OSHA 7 See: NMAM or OSHA Methods		
Personal Protection & Sanitation (See protection) Skin: Prevent skin contact Eyes: Prevent eye contact Wash skin: When contaminated Remove: When wet (flammable) Change: No recommendation		First Aid (See procedures) Eye: Irrigate immediately Skin: Soap wash promptly Breathing: Respiratory support Swallow: Medical attention immediately
Important additional information about respirator selection		
Respirator Recommendations NIOSH/OSHA Up to 2000 ppm: <ul style="list-style-type: none"> (APF = 25) Any supplied-air respirator operated in a continuous-flow mode[£] (APF = 25) Any powered, air-purifying respirator with organic vapor cartridge(s)[£] (APF = 50) Any chemical cartridge respirator with a full facepiece and organic vapor cartridge(s) (APF = 50) Any air-purifying, full-facepiece respirator (gas mask) with a chin-style, front- or back-mounted organic vapor canister (APF = 50) Any self-contained breathing apparatus with a full facepiece (APF = 50) Any supplied-air respirator with a full facepiece 		
Emergency or planned entry into unknown concentrations or IDLH conditions: <ul style="list-style-type: none"> (APF = 10,000) Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode (APF = 10,000) Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained positive-pressure breathing apparatus 		
Escape: <ul style="list-style-type: none"> (APF = 50) Any air-purifying, full-facepiece respirator (gas mask) with a chin-style, front- or back-mounted organic vapor canister/Any appropriate escape-type, self-contained breathing apparatus 		
Exposure Routes inhalation, ingestion, skin and/or eye contact		
Symptoms Irritation eyes, respiratory system; central nervous system depression		
Target Organs Eyes, respiratory system, central nervous system		
See also: INTRODUCTION See ICSC CARD: 0436		



NIOSH Pocket Guide to Chemical Hazards

Methyl chloroform		CAS 71-55-6
<chem>CH3CCl3</chem>		RTECS KJ2975000
Synonyms & Trade Names Chlorothene; 1,1,1-Trichloroethane; 1,1,1-Trichloroethane (stabilized)		DOT ID & Guide 2831 160
Exposure Limits NIOSH REL: C 350 ppm (1900 mg/m ³) [15-minute] See Appendix C (Chloroethanes) OSHA PEL†: TWA 350 ppm (1900 mg/m ³)		
IDLH 700 ppm See: 71556		Conversion 1 ppm = 5.46 mg/m ³
Physical Description Colorless liquid with a mild, chloroform-like odor.		
MW: 133.4	BP: 165°F	FRZ: -23°F
VP: 100 mmHg	IP: 11.00 eV	Sp.Gr: 1.34
Fl.P: ?	UEL: 12.5%	LEL: 7.5%
Combustible Liquid, but burns with difficulty.		
Incompatibilities & Reactivities Strong caustics; strong oxidizers; chemically-active metals such as zinc, aluminum, magnesium powders, sodium & potassium; water [Note: Reacts slowly with water to form hydrochloric acid.]		
Measurement Methods NIOSH 1003 See: NMAM or OSHA Methods		
Personal Protection & Sanitation (See protection) Skin: Prevent skin contact Eyes: Prevent eye contact Wash skin: When contaminated Remove: When wet or contaminated Change: No recommendation		First Aid (See procedures) Eye: Irrigate immediately Skin: Soap wash promptly Breathing: Respiratory support Swallow: Medical attention immediately
Important additional information about respirator selection Respirator Recommendations NIOSH/OSHA Up to 700 ppm: (APF = 10) Any supplied-air respirator* (APF = 50) Any self-contained breathing apparatus with a full facepiece Emergency or planned entry into unknown concentrations or IDLH conditions: (APF = 10,000) Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode (APF = 10,000) Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained positive-pressure breathing apparatus Escape: (APF = 50) Any air-purifying, full-facepiece respirator (gas mask) with a chin-style, front- or back-mounted organic vapor canister/Any appropriate escape-type, self-contained breathing apparatus		
Exposure Routes inhalation, ingestion, skin and/or eye contact		
Symptoms Irritation eyes, skin; headache, lassitude (weakness, exhaustion), central nervous system depression, poor equilibrium; dermatitis; cardiac arrhythmias; liver damage		
Target Organs Eyes, skin, central nervous system, cardiovascular system, liver		
See also: INTRODUCTION See ICSC CARD: 0079 See MEDICAL TESTS: 0141		

ATTACHMENT B

PERSONNEL ACKNOWLEDGEMENT RECORD

ATTACHMENT B
PERSONNEL ACKNOWLEDGMENT RECORDS

CCA Employees

Project staff must sign the master copy of this document, indicating they have read and understand it. The employee's signature indicates acceptance and compliance with the requirements of the SSHP. Copies of this document must be made available for their review and readily available at the job site.

LOG OF CCA PROJECT PERSONNEL

ATTACHMENT B
PERSONNEL ACKNOWLEDGMENT RECORDS

LOG OF CCA PROJECT PERSONNEL

APPENDIX B
PERSONNEL ACKNOWLEDGMENT RECORDS

Visitors

It is CCA's policy that visitors must furnish their own PPE. Visitors are required to sign the Visitor Log and comply with guidelines, rules, and procedures presented herein. If the visitor represents a regulatory agency concerned with site safety and health issues, the SSHO must immediately notify the DSHO.

VISITOR LOG

APPENDIX B
PERSONNEL ACKNOWLEDGMENT RECORDS

Safety and Health Meetings

Project personnel must receive initial safety and health orientation. Thereafter, a brief tailgate safety meeting is required as deemed necessary by the SSHO. Health and safety meetings will be held at least once every week or when risks and/or hazards change.

SAFETY AND HEALTH MEETING LOG

ATTACHMENT C

ACCIDENT INVESTIGATION FORM



ACCIDENT REPORT FORM

Employee Name:	Date of Birth:
Home Address:	Phone: ()
Sex: <input type="checkbox"/> Male <input type="checkbox"/> Female	Job Title:
Office: <input type="checkbox"/> Phoenix <input type="checkbox"/> Tucs <input type="checkbox"/>	Date of Hire:
Hours Usually Worked: Hours per day:	Hours per week:
Total hours worked:	

Where did the accident or exposure occur (include address)?

County: On employer's premises? Yes No
What was the employee doing when the injury occurred (Be specific):

Fully describe how the accident or exposure occurred (use addition paper if necessary):

What could be done to prevent such another similar accident or exposure?

Object or substance that directly injured employee:

Describe the injury or illness: Part of body affected:
Name and address of physician:
If hospitalized, name and address of hospital:
Date of injury/illness: Time of day: Loss of one or more days of work
 Yes No If yes, last day worked:
Has employee returned to work? If yes, date returned:
Did employee die? If yes date of death:

Completed by (Print): Signature:
Title: Date:

An accident/exposure report must be completed by the supervisor or site safety officer immediately upon learning of an accident or incident. The completed report must be transmitted to the Office Manager and Health and Safety Manager immediately upon completion.

ATTACHMENT D

FIRST AID AND EMERGENCY CARE

Most accidents occurring at a job site will require minimal first aid available through use of the first aid kit(s) at the work site or in the support facility. For more serious medical emergencies that may or may not require professional medical attention, the American National Red Cross (1988) has developed first-aid procedures that can be followed until professional medical attention is obtained. The following sections present a summary of these procedures.

HEAT EMERGENCIES

There are three forms of heat emergencies: heat stroke, heat exhaustion, and heat cramps. Of these three, **heat stroke** is the most serious because it is life-threatening.

HEAT STROKE

Symptoms

- Hot, red skin
- Very small pupils
- Very high body temperature
- Skin may feel dry

First Aid

- Call for medical assistance.
- Move the victim to a cool (not cold) place immediately.
- Cool the victim quickly by immersing him/her in a cool (not cold) bath, wrapping wet sheets around the victim and fanning him/her, or spraying the victim with cool water.
- Monitor the victim for shock (pages D-7 and D-8) until medical assistance arrives.

HEAT EXHAUSTION

Symptoms

- Cool, pale, and moist skin
- Heavy sweating
- Dilated pupils
- Headache
- Nausea
- Dizziness
- Vomiting
- Normal body temperature

First Aid

- Move the victim out of the heat.
- Have the victim lie down with feet elevated.
- Loosen or remove the victim's clothes.
- Cover the victim with wet towels or sheets or apply cold packs wrapped in cloth.
- Fan the victim.
- Have the victim drink one-half glass of water every 15 minutes if they are conscious and able to keep the fluid down.

HEAT CRAMPS

Symptoms

- Muscular pains and spasms

First Aid

- Move the victim out of the heat.
- Have the victim drink one-half glass of water every 15 minutes for one hour.

COLD EMERGENCIES

Severe cold exposure can be an immediate danger to life and health. The two most serious forms of cold exposure are hypothermia and frostbite.

HYPOTHERMIA

Symptoms

- Shivering
- Dizziness
- Numbness
- Confusion
- Weakness
- Impaired judgment
- Impaired vision
- Drowsiness

Stages

- Shivering
- Apathy
- Loss of consciousness
- Decreasing pulse rate and breathing rate
- Death

First Aid

- Call for medical assistance.
- Move the victim to a warm place.
- Remove the victim's wet clothing, as applicable.
- Cover the victim with a dry blanket.
- Warm the victim slowly.
- Monitor the victim's breathing and heart rate.
- Give the victim warm broth or water - no alcohol or caffeine.

FROSTBITE

Symptoms

- Area is very cold to the touch and numb
- Slightly flushed skin

- Mild frostbite will appear on the edges of appendages as white or grayish-yellow with hardened skin
- Moderate frostbite will show a larger portion of the appendages as white or grayish-yellow and skin will have blistered
- Severe frostbite is grayish-blue and skin will be hard, cold, and numb. There is a danger of gangrene developing from severe frostbite

First Aid

- Move the victim to a warm place.
- Place the frostbitten areas in warm (not hot) water.
- Handle the frostbitten areas gently.
- Do not rub, massage, or apply unnecessary pressure to the frostbitten area.
- Place dry gauze between frostbitten toes or fingers.
- Bandage frostbitten areas loosely.

ANIMAL BITES

Infection from an animal bite can develop quickly: first aid should be administered immediately.

First Aid

- Control the bleeding.
- Gently wash the wound unless bleeding heavily.
- Cover the bite with a bandage.
- Have the victim see a trained medical person.

RABID ANIMAL BITES

Rabies can be found in the saliva of skunks, bats, raccoons, cattle, cats, dogs, foxes, squirrels, prairie dogs, rats, and mice.

First Aid

- Observe the animal for unusual behavior.
- Get the victim to medical care.
- Give a description of the animal and where it was last seen to the police and/or animal control so they can capture the animal for determination of rabies infection.
- Do NOT attempt to capture or restrain the animal yourself.

INSECT BITES AND STINGS

Inset bites and stings may be tolerated by some individuals more so than others. A past history of bite and sting tolerance is not indicative of continued tolerance. All bite and sting victims should be monitored for allergic reactions.

The following is a summary of symptoms and first-aid response for an allergic reaction to a bite or sting.

Symptoms

- Pain
- Swelling of the bite or sting area, which may be accompanied by swelling of the throat
- Redness or discoloration of the bite or sting area
- Itching

- Hives
- Decreased awareness
- Breathing noisy or difficult

First Aid

- Remove the stinger with tweezers or scrape with a rigid item without squeezing it (which may release more venom).
- Wash the area of the bite or sting.
- Place a cold pack wrapped in cloth on the area.
- Keep the bite or sting below heart level.
- Get the victim to medical assistance if an allergic reaction is observed.

SNAKE BITES

Few people actually die from snake bites; however, quick response to a snake bite is imperative.

First Aid

- Call for medical assistance.
- Immobilize the bitten area.
- Keep the bitten area below the heart level.
- Keep the victim calm and still.
- Observe victim for symptoms of shock (pages D-7 and D-8).
- Do not cut above the bite and aspirate the poison.
- Do not use a tourniquet.
- Give a description of the snake to the medical responder.

SEVERE BLEEDING

First Aid

Stop external bleeding by:

- Applying direct pressure to the wound using a clean cloth.
- Apply cloths on top of the first one if bleeding persists; do not remove original cloth.
- If there is no fracture, raise the wound above the level of the heart.
- Apply pressure at the appropriate pressure point (squeezing the main artery against the bone in the forearm or against the pelvis in the groin) while continuing pressure on and elevation of the wound.
- Wrap the wound using subtle pressure to tighten the wrap.
- Check for a pulse on the injured limb to determine that the wrap is not too tight.
- Call for medical assistance.

INTERNAL BLEEDING

Internal bleeding may be as innocuous as a bruise to a condition that threatens life and health.

Symptoms

- Tender, bruised, swollen or rigid abdomen
- Fractured ribs or pelvis
- Vomiting small to large amounts of blood

- Injuries that have penetrated the body cavity
- Rectal or vaginal bleeding
- Difficulty breathing
- Pulse rate is abnormal
- Cool, moist skin
- Pallor

First Aid

- Treat small bruises by applying a cold pack to the injury.
- Obtain medical help immediately if more severe internal bleeding is suspected.
- Observe the victim's breathing and monitor his/her pulse.
- Keep the victim calm and still.
- Loosen the victim's clothing.
- Place the victim on his/her side if vomiting.
- Monitor the victim for symptoms of shock.

SHOCK

Shock can be caused by internal and external bleeding, insect bites or stings, snake bites, electrical shocks, severe injuries or burns, as well as other medical conditions. First aid and medical assistance is imperative for shock victims because shock is caused by a lack of sufficient blood supply to such vital organs as the heart, the lungs, and the brain.

Symptoms

- Confused behavior
- Either very slow or very fast pulse rate
- Either fast, shallow breathing or very slow breathing
- Weak and trembling limbs
- Cool, moist skin
- Pallor or bluish skin
- Pupils are dilated

First Aid

- Improve victim's circulation by lying them down with feet elevated if there are no leg fractures or suspected neck/head injuries. (Lay the victim flat if injuries are suspected.)
- If no injuries are suspected, a semi-reclining position may be used to alleviate breathing problems.
- If the victim is vomiting turn him/her onto their side.
- Keep the victim warm.
- Call for medical assistance.
- Monitor the victim's heart rate and breathing.

VICTIM NOT BREATHING

First Aid

- Tap or gently shake the victim to see if there is a response. Ask, "Are you okay?"
- Roll the victim onto his/her back and toward you.

- Tilt the head back while lifting the chin.
- Check for breathing for 3 to 5 seconds.
- Pinch the nose shut, seal your mouth over the victim's mouth and give two 1- to 1-1/2-second breaths while keeping the head tilted back.
- Check for a pulse.
- Call or send someone for help.
- Continue rescue breathing, if necessary, by breathing into the victim's mouth for 1 to 1-1/2 seconds every 5 seconds.
- Observe victim for a pulse approximately every minute.

VICTIM NOT BREATHING AND HAS NO PULSE

First Aid

- Roll the victim onto his/her back and toward you.
- Tilt the head back while lifting the chin.
- Check for breathing for 3 to 5 seconds.
- Pinch the nose shut, seal your mouth over the victim's mouth and give two 1- to 1-1/2-second breaths while keeping the head tilted back.
- Check for a pulse.
- Call or send someone for help.
- Locate the notch at the lower end of the breastbone.
- Place the heel of your hand two fingers-width up from the end of the notch.
- Place your other hand on top keeping the fingers of your hands off the chest.
- Position your shoulders directly over your hands.
- Using a steady, firm force, bending at the waist, compress the breastbone 1-1/2 to 2 inches for 15 counts in 10 seconds.
- Perform rescue breathing (2 quick breaths as above).
- Repeat this for a total of 4 cycles.
- Recheck pulse.
- Continue cardiopulmonary resuscitation (CPR) procedures as described above until medical assistance arrives.

BURNS

There are four types of burns: heat burns, chemical burns, electrical burns, and radiation burns. Each type has three categories of burns: first degree, second degree, and third degree.

First Degree Burn Symptoms

- Least severe
- Skin will be red or discolored
- Mild swelling
- Pain

Second Degree Burn Symptoms

- Burn extends deeper into the skin
- Skin is red or mottled

- Blistering
- May appear wet from skin fluid loss
- Painful

Third Degree Burn Symptoms

- Deepest burn; extends through all skin layers
- Skin appears white or charred
- Can look like second-degree burns
- Pain may be severe or, if nerve endings are destroyed, may not occur at all
- Can occur in patches with less severe burns

First Aid for Heat Burns

- Flush with cool running water if there are no blisters or charring. Apply moist dressings and bandage loosely.
- If blisters or charring are present, apply a dry dressing and bandage loosely. Do not use water.
- Call for medical assistance.

First Aid for Chemical Burns

- Flush the chemicals from the skin with lots of water.
- Continue flushing for 15 to 30 minutes.
- Remove any contaminated clothing or jewelry.
- Cover burns loosely with a dry bandage or dressing.
- Call for medical assistance.

First Aid for Electrical Burns

- Avoid contact with electrical source.
- Shut down the electrical source.
- Cover all burns with a loose dry dressing and bandage.
- Provide care for shock (pages D-7 and D-8) as needed.
- Call for medical assistance.

First Aid for Radiation Burns

- None.
- Decontaminate the victim.
- Obtain medical assistance immediately.

EYE INJURIES

Eye injuries should always be treated as a serious injury.

Symptoms

- Visible foreign object
- Redness
- Burning
- Pain
- Headache
- Tearing

First Aid

- Use care and be gentle when touching the eyes.
- Wash hands before caring for an eye injury, if possible.
- If an object is in the eye, lift the upper eyelid, have the victim look down and flush the eye with clean water or eye wash solution.
- If there are chemicals in the eye, flush the eye with clean water or eye wash solution from the nose outward for 15 to 30 minutes.
- For objects in the eye (whether removed through flushing or not) and for chemicals in the eye, wrap a bandage loosely around both eyes.
- If the eye is cut or there is a penetrating object in the eye, place a cup over the injured eye and wrap both eyes loosely with a bandage. Do not attempt to remove the penetrating object.
- Obtain medical assistance for all (even minor) eye injuries.

NOSE INJURIES

Nose injuries can be indicative of more serious injuries to the head, back, or neck. Caution should be used to assess this type of injury. Nosebleeds are typically a less serious injury but can be severe enough to cause shock from loss of blood. Be sure to ask the victim how the nosebleed began.

First Aid

- Have the victim sit down.
- Have him/her lean forward with the chin resting on the chest.
- Pinch the nose.
- Keep the victim calm and quiet until the bleeding has stopped.

Symptoms of a More Serious Nose Injury

- Swelling and pain
- Pupils dilated unevenly
- Bloody or clear fluid draining from either the ears or the nose
- Loss of feeling and movement in appendages

First Aid for a More Serious Nose Injury

- Do not attempt to stop the flow of fluid from the nose.
- Keep the victim's head and neck stable.
- Keep the victim calm and quiet.
- Call for medical assistance.

FRACTURES

There are two types of fractures: simple (one internal fracture) and compound (two or more fractures often breaking the skin). The compound fracture is more serious because of the accompanying open wound. Fractures occurring in the body may be indicative of internal injuries.

Symptoms

- A grating sensation and/or a snapping sound when the appendage is moved

- Deformities
- Pain and tenderness
- Bruising and swelling
- Immobility of the injured part

Note: First aid for fractures, dislocation, sprains, and strains are similar for these injuries. The first aid for these injuries will be discussed after the symptoms.

DISLOCATIONS

Symptoms

- Deformity
- Swelling and tenderness
- Pain in the joint
- Loss of or limited movement

SPRAINS OR STRAINS

Sprains are the result of stretched or torn tendons or ligaments around the joints. Torn muscles are indicative of strains.

Symptoms

- Pain in the joint
- Sharp pain
- Tender to the touch
- Bruising and swelling
- Stiffness

First Aid for Fractures, Dislocations, Sprains, or Strains

- If the injury is to the head, neck, or back, stabilize the head and neck. Do not attempt to move the victim unless absolutely necessary. Obtain medical assistance immediately. Keep the victim calm and quiet.
- Determination of the precise injury is often difficult, so remember this rule of thumb: "When in doubt, splint."
- Splint only if it can be done without causing more pain and discomfort to the victim.
- The injury must be splinted in the position in which it is found. Do not attempt to straighten the injured part.
- Splint the injured area as well as the surrounding joints so that the entire limb is immobilized.
- Check for a pulse before and after splinting.
- Call for medical assistance.